Tappi

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JULY 1959

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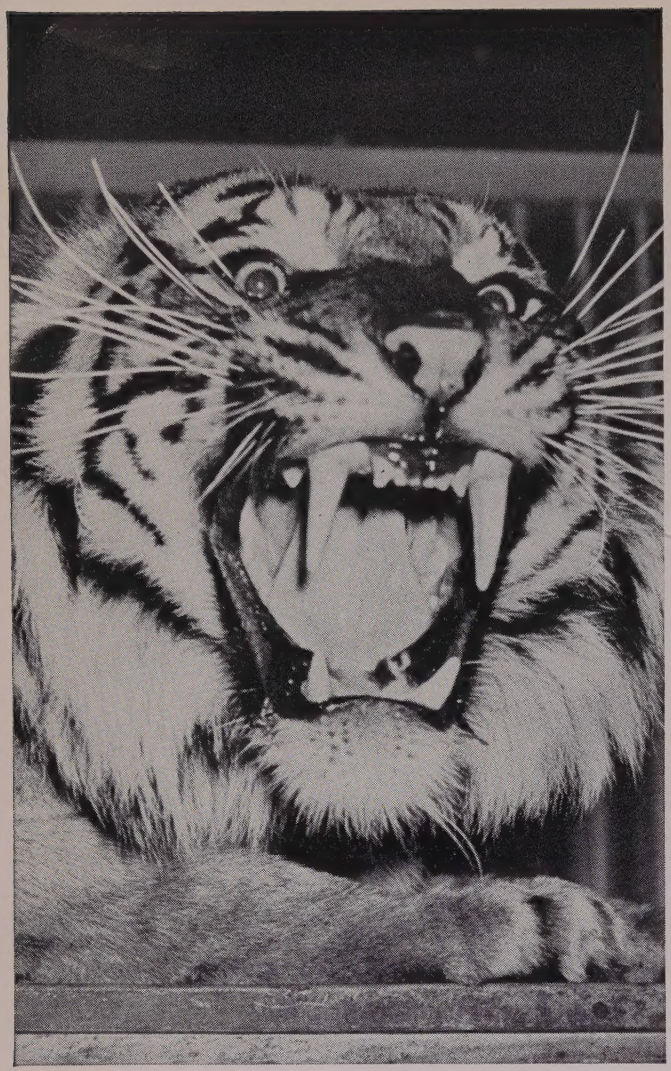

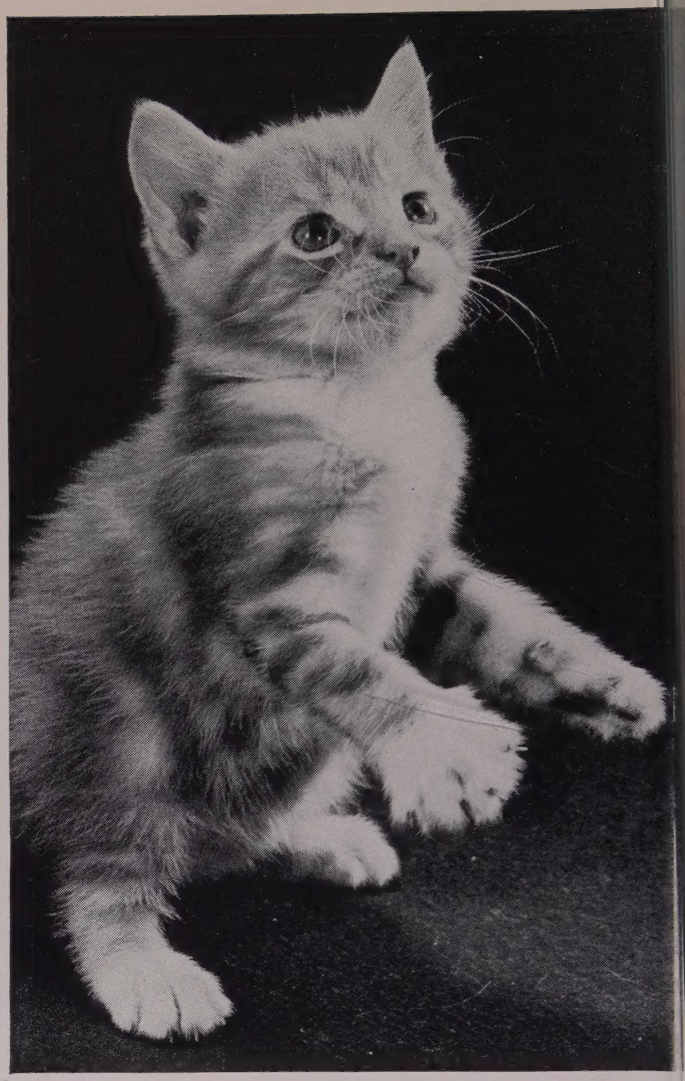

They're both cats, but...

each is in a class by itself . . . like Wyandotte's PURECAL® O

You'd never mistake a jungle tiger for a pet kitten . . . but are you as aware of the difference in calcium carbonates?

For, all calcium carbonates are *not* alike. Wyandotte PURECAL O – the calcium carbonate that's in a class by itself – is precipitated by a special process which yields agglomerate-free particles 0.15 micron in size, consistently cubical in shape. What's more, PURECAL O exceeds U.S.P. purity standards – and is a product of *exceptional* whiteness.

For these reasons, PURECAL O in paper coatings improves flow and leveling for better printability . . . provides a smoother, whiter surface that brings out all the beauty of the four-color process, and reproduces halftones in faithful detail.

Try PURECAL O in your formulation, and discover for yourself how it can help you make a *better* sheet to sell at *competitive* prices. Write for samples and technical data, today. *Wyandotte Chemicals Corporation, Dept. 754-T, Wyandotte, Michigan. Offices in principal cities.*

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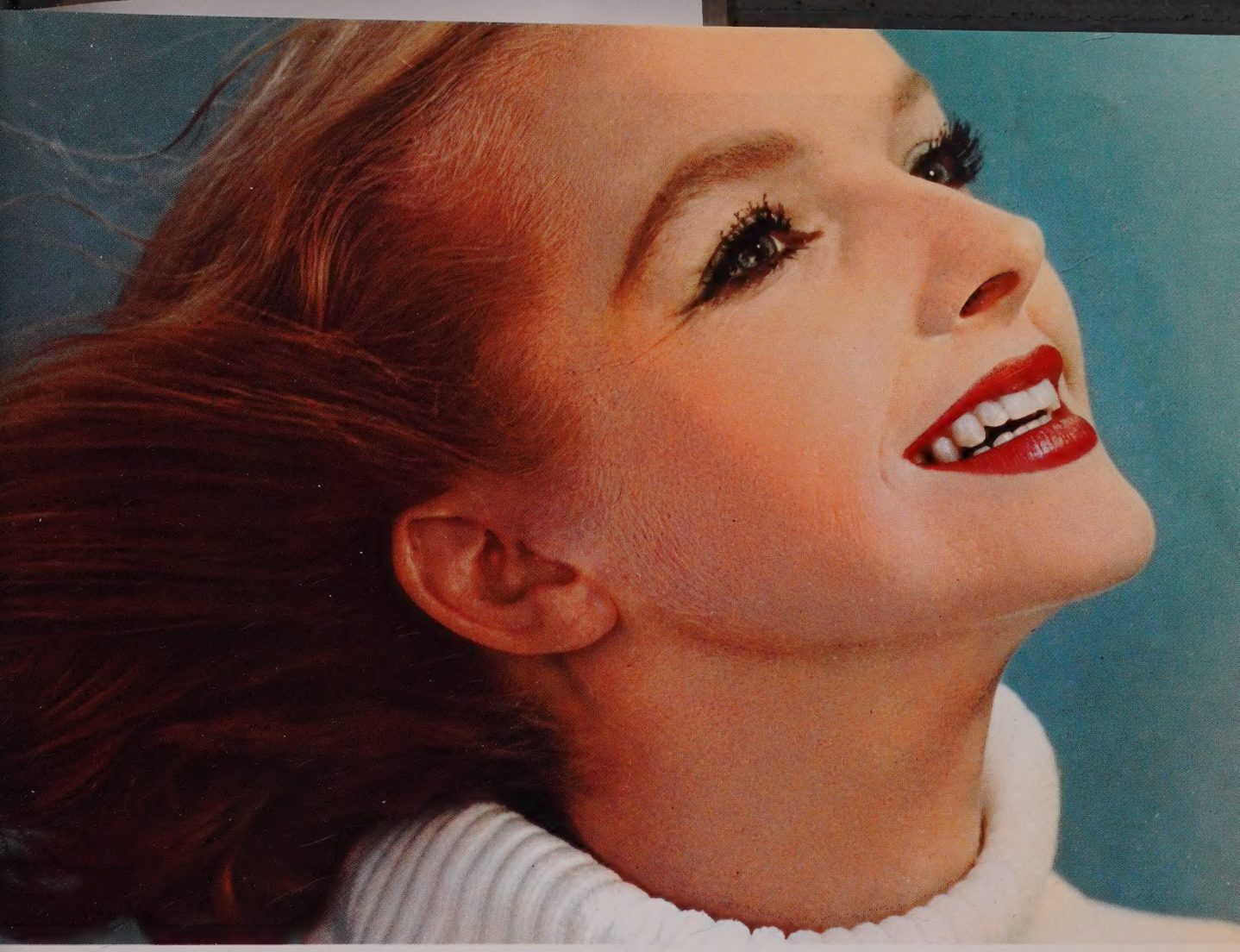

Alpha® Protein...

to get all that the camera saw...on paper

All the alluring beauty first captured by the camera is faithfully recorded here . . . by the unexcelled quality of Alpha Protein-processed paper. Fidelity of reproduction is reflected in each strand of hair, each subtle flesh tone.

Every day, more coating mill operators are specifying Alpha Protein—the largest-selling adhesive for high solids coated offset paper. Reason: they want the controlled product quality, faster machine speeds, higher coat weights, reduced costs—and better-satisfied customers!

Alpha Protein extra high solids coatings permit faster machine speeds with production upped 15 percent in many cases. Investigate the application of Alpha Protein in your coating operation—it can pay you well!

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Acorn of the Eastern Red Oak

Best seller...1989

This acorn may grow to be a tree which will be the source of paper for a best-selling book. It is likely that the paper will bleach whiter, and *stay* whiter—thanks to hydrogen peroxide from Shell Chemical.

In chip-groundwood pulp bleaching, hydrogen peroxide gives high brightness plus markedly increased reversion resistance. For, unlike other bleaches, the *higher* you bleach with hydrogen peroxide, the *greater* the resistance to reversion.

Other Shell chemicals, listed in the column at the right, help the paper and packaging industries produce better products that build lasting customer satisfaction.

The Shell Chemical Technical Service Laboratory—one of the nation's best equipped—will gladly help you solve your paper problems, from pulping to conversion. For prompt assistance, samples or technical literature, write to your nearest Shell Chemical district office.

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- Glycerine
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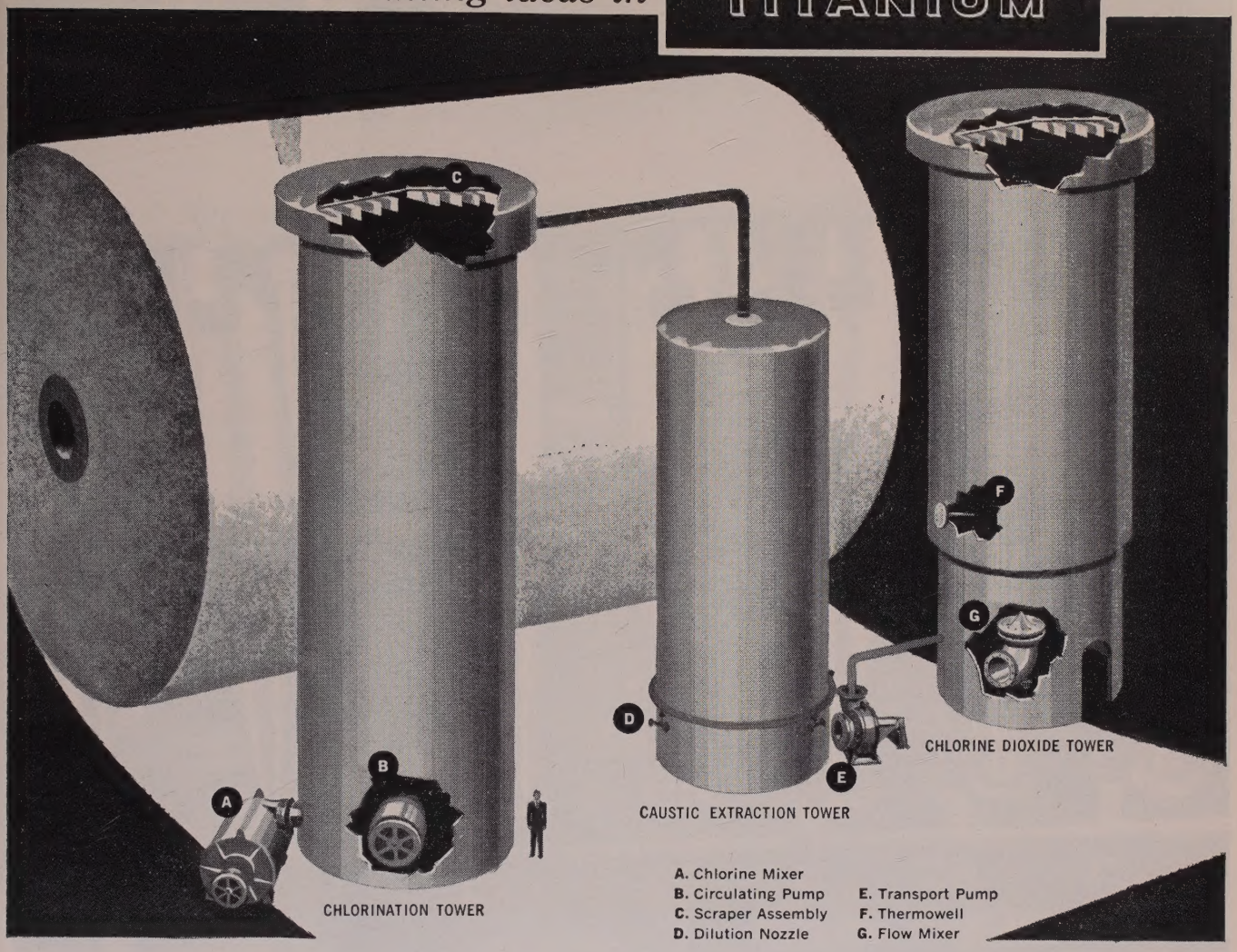
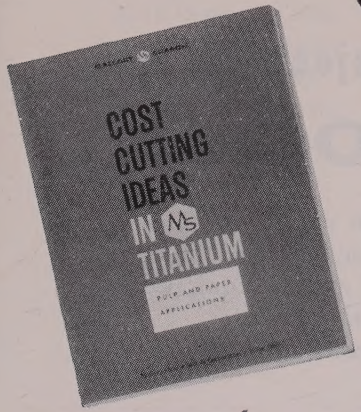
Cost cutting ideas in **TITANIUM**



TITANIUM BELONGS IN THIS CORROSIVE PROCESSING PICTURE



***Write for** Technical Data Sheets on "Cost Cutting Ideas in Titanium for the Pulp and Paper Industry"—on your business letterhead, please. Address: Commercial Market Development, Dept. C, Mallory-Sharon Metals Corporation, Niles, Ohio.*

Here's why: Today, titanium is competitive pricewise with nickel base alloys when density is taken into account. Its service life is often many times that of nickel base alloys or stainless steel. And titanium is about the same to weld and fabricate.

The corrosion resistance of titanium in chlorine dioxide and hypochlorite solutions is outstanding. No wonder it is rapidly finding new applications in the pulp and paper and chemical industries.

Want proof? A large pulp processor used titanium orifice plates in chlorine dioxide reactors for over a year, with no evident corrosion. The best alternate material lasted only 2 weeks.

At a large chemical plant, commercially pure titanium specimens immersed for 28 days in a calcium hypochlorite storage tank showed an average corrosion rate of less than .10 mils per year.

Mallory-Sharon is ready to assist you *now* in applying titanium's outstanding corrosion resistance in pulp and paper processing . . . or other corrosive applications. Send for the helpful information illustrated at the left.

MALLORY SHARON

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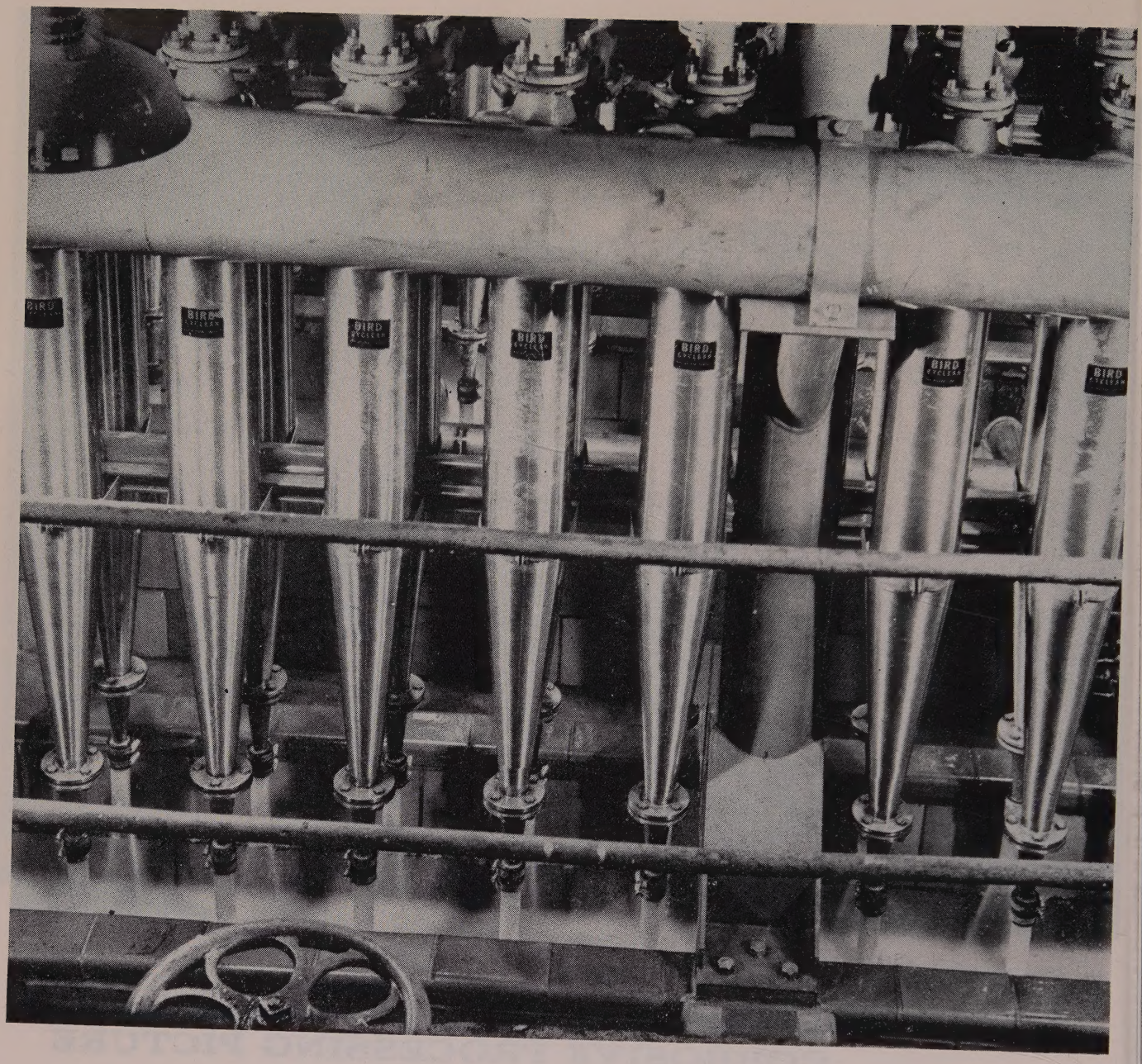
Integrated producer of Titanium · Zirconium · Special Metals



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CYCLEAN YOUR STOCK

Bird Cycleans are proving extraordinarily effective in removing shives and specks, improving fibre hydration, reducing breaks, increasing wire life and helping to maintain maximum output of uniformly clean paper.

Cycleans are available in units of 4″ dia., 36 gpm; 7″ dia., 120 gpm; 12″ dia., 500 or 850 gpm. The 4″ units may be equipped with patent, non-plugging purge tips.

Ask us to make recommendations, layouts and estimates.

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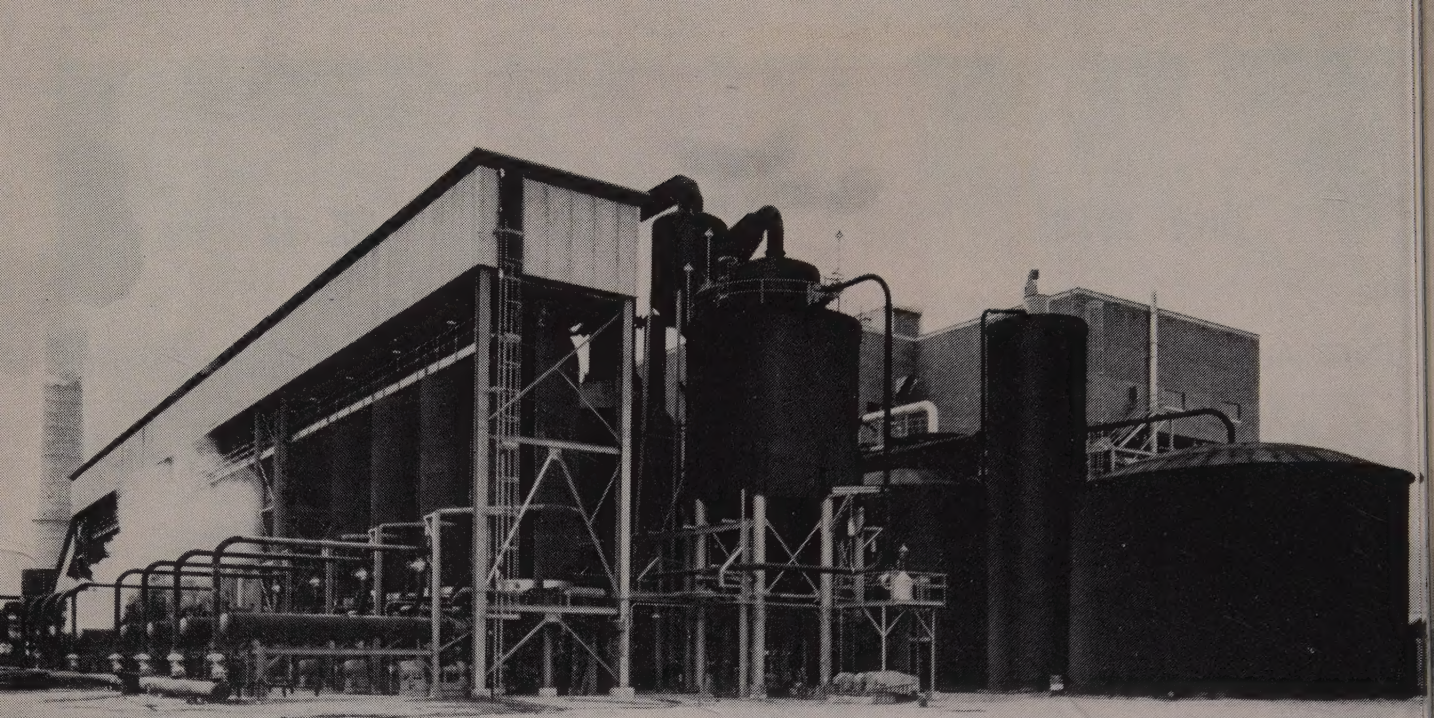

LUMMUS ENGINEERS AND CONSTRUCTS

Buckeye Cellulose Corporation's Wood Pulp Expansion

NATION'S OLDEST PRODUCER OF CELLULOSE PULP DOUBLES PRODUCTION CAPACITY OF FOLEY, FLORIDA, PLANT. The Buckeye Cellulose Corporation, America's oldest yet fastest growing cellulose pulp producer, will soon put into operation a new $20,000,000 wood pulp unit at Foley, Florida, thus completing an expansion program which doubles the plant's original production capacity.

The Lummus Company engineered and constructed the process units for these expanded facilities which assure Buckeye's customers a steady supply of highest quality Southern pine pulp. This new Lummus-designed plant is still another step in the growth of a company with 37 years of experience in producing cellulose pulp. Buckeye has the world's most complete line of celluloses, being the only producer of cellulose from both wood and cotton linters. Its cotton linter pulp mill was established in Memphis, Tennessee, in 1921.

Highest standards of processing are demanded by Buckeye to provide the best in cleanliness, brightness, strength and other specific qualities desired by users of Buckeye pulp. Long a research-minded company, Buckeye spends a higher percentage of sales on research activities than any other pulp producer.

In addition to engineering and constructing the process units for the Foley expansion, Lummus hired and coordinated various subcontractors for other portions of the plant. In spite of the complexity of such a large program, Lummus met the construction schedule and the new mill is assured a smooth start-up.

The Lummus Company has designed, engineered and constructed over 800 complete plants for the process industries throughout the world in the last 50 years. Lummus world-wide experience is at your disposal.

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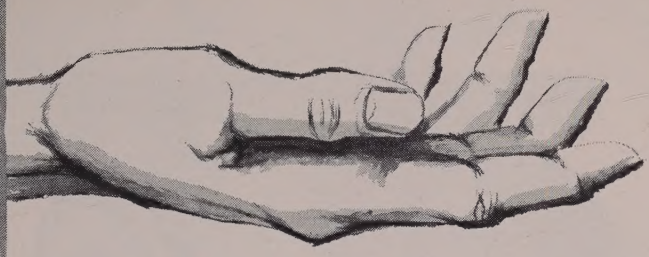
Flip a handful of Westvaco Soda Ash downwind. No rising cloud here! Do the same with any ordinary soda ash. See the difference?



this is all the equipment

you need to prove

Westvaco® Soda Ash

dusts less . . . flows more freely!

The Cupped-hand Test

Let the roller-shaped crystals of Westvaco Soda Ash pour from your palm. Try another soda ash. Note how much faster and more smoothly Westvaco Soda Ash flows.

You can readily see the difference in physical characteristics that makes Westvaco Soda Ash unique —faster and cleaner to unload and to handle.

Simple comparative dissolution tests show it's quicker to dissolve . . . and to produce solutions with crystal clarity. Chemical analyses tell you it's purer, too . . . low in iron, salts, heavy metals and insolubles . . . and entirely ammonia-free!

Only a plant trial, however, can prove how Westvaco Soda Ash can simplify and speed up your processing and better your end-products.

If you want to make a few pre-trial tests, we'll gladly supply samples. But be sure to schedule practical in-use tests, too, and see what Westvaco Soda Ash can do for you. Why not place a trial order now?

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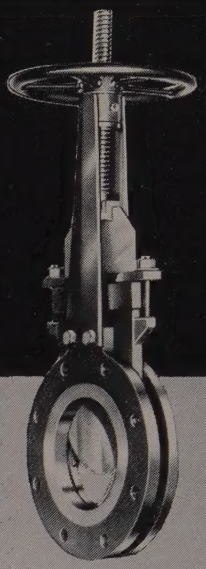

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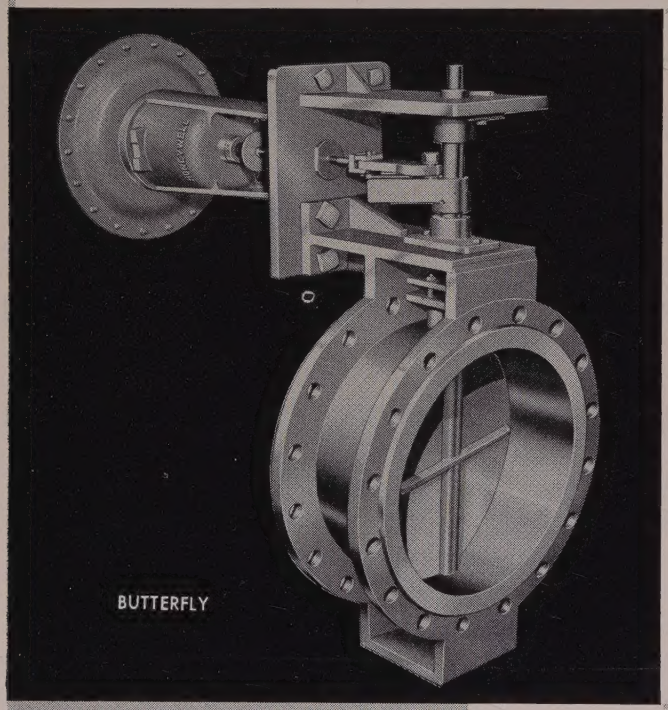

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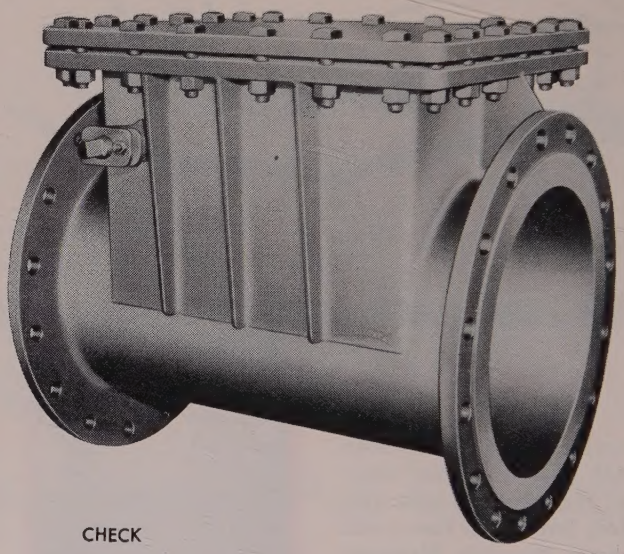
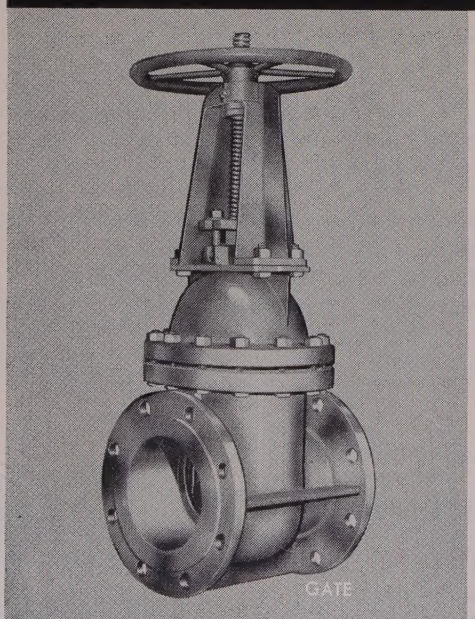


CHECK



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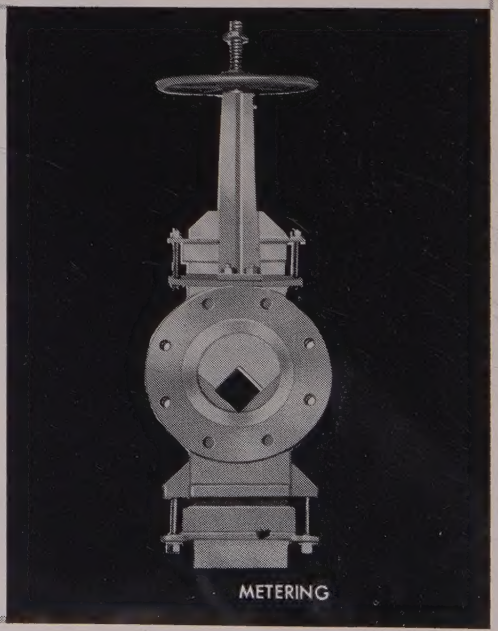

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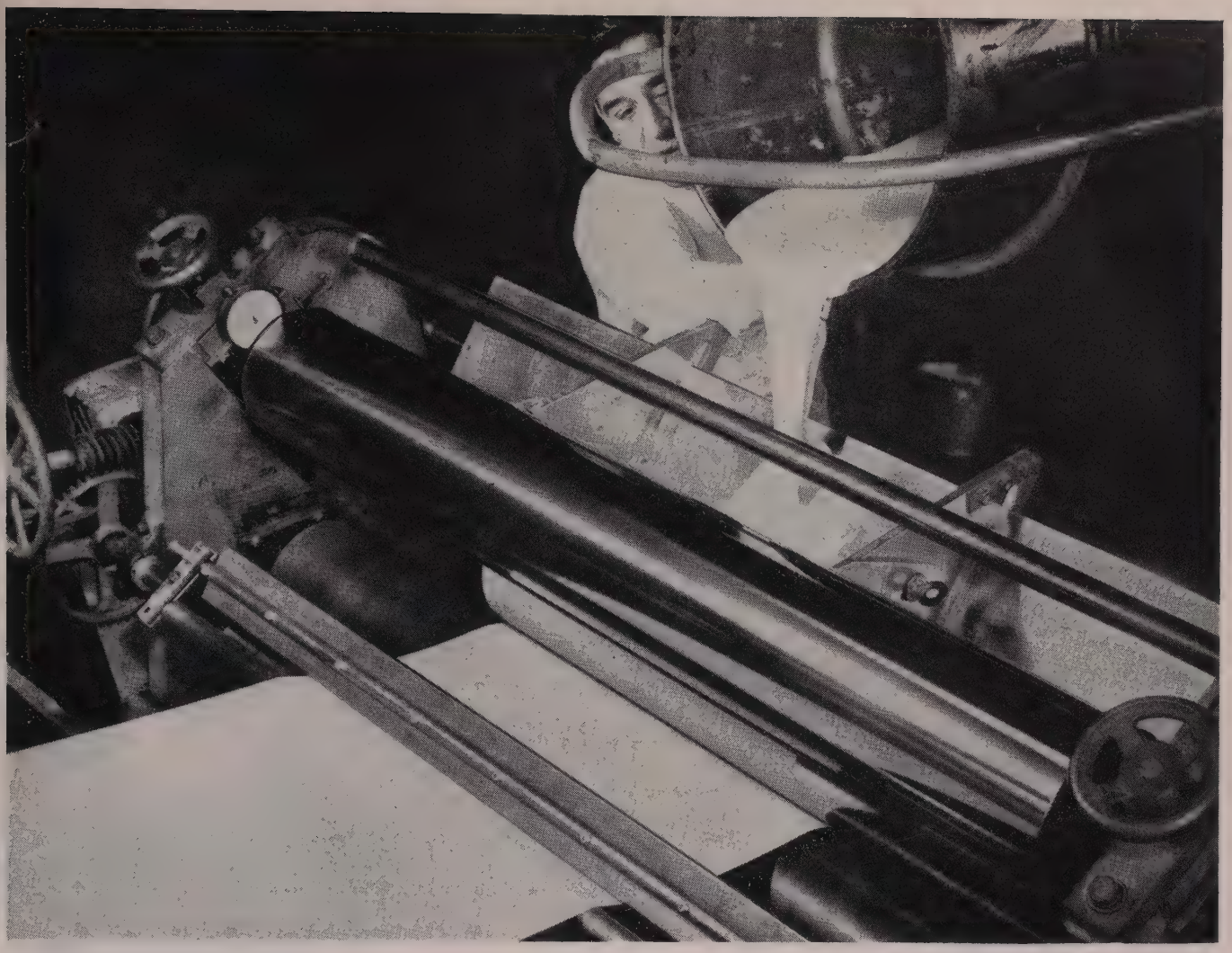


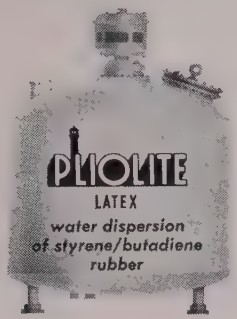
Want Bigger Dividends from Paper Stocks?

The big pay-off in paper comes when a manufacturer supplies a specific need with a new and superior product. A recent example: plasticized printing paper, a combination of paper fibers, and a CHEMIGUM LATEX by Goodyear. Perhaps you're working on a development of comparable importance right now. Could be a paper with exceptional wet-strength – or a unique texture –or unprecedented fold qualities.

Whatever your project, it will pay you substantial dividends to explore the advantages of Goodyear's three basic latices: PLIOLITE LATEX (water dispersion of styrene-butadiene rubber), PLIOVIC LATEX (water dispersion of vinyl copolymer resin), and–of course –CHEMIGUM LATEX (water dispersion of nitrile rubber). All three are available in a broad range of monomer ratios, particle sizes and types of stabilization. They can be used alone–or in combination with each other–or as modifiers of your present materials.

Among the benefits you can expect from these latices: superior oil- and grease-resistance, improved folding and bending qualities, excellent printing surface, high edge tear, added burst strength, better feel and drape.

You can also expect the finest in technical assistance when you specify a Goodyear latex. Contact your Goodyear Chemical Division Representative for all the facts. Or write for details–including latest *Tech Book Bulletins*–to Goodyear, Chemical Division, Dept. G-9432, Akron 16, Ohio.



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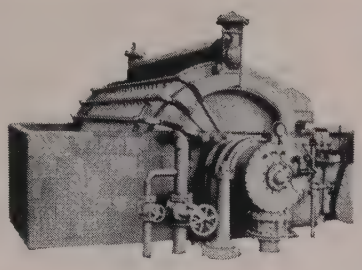
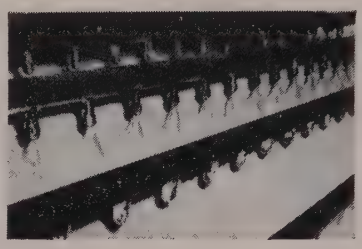
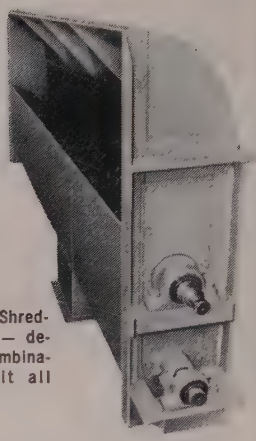


Oliver Vacuum Bleach Washer — now available in advanced designs and materials.



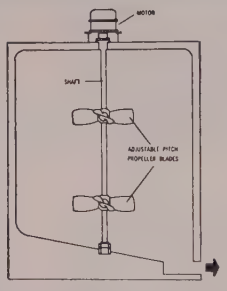
Dorr-Oliver Escoe and Nycoe Nozzles — minimize channeling and foam in the sheet.



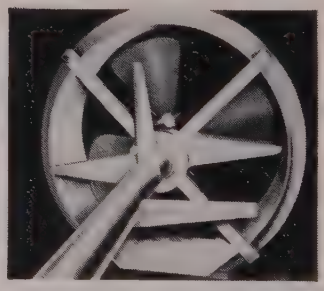
Dorr-Oliver Shredder-Conveyor — designs and combinations to suit all installations.

AGITATORS

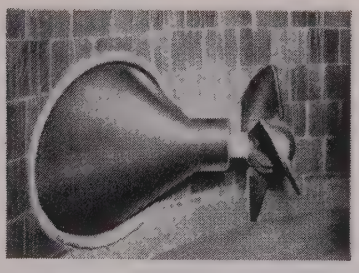
Dorr-Oliver manufactures a variety of agitators specifically engineered to meet the specialized requirements of every operation in the bleaching system.



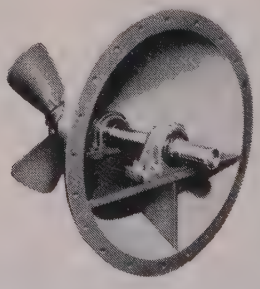
Dorr-Oliver Blending Chest Agitators — designed for proper agitation with minimum power requirements.



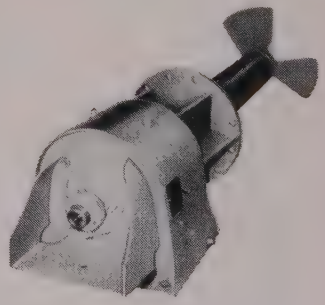
Dorr-Oliver Horizontal Chest Agitators — a complete line designed for tub or mid-feather type chests.



Dorr-Oliver Bleach Tower Agitators — tangentially mounted with either fixed or fully adjustable blades.



Dorr-Oliver Tower Agitators — for high density storage towers. Adjustable propellers.

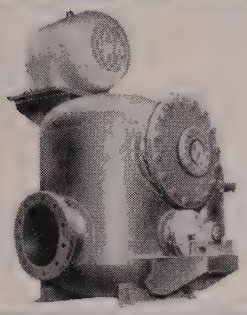


Falk All-Motor Driven Agitator — single unit motor, reducer and propeller assembly.

BLEACH PLANT EQUIPMENT

pulp mill engineers for pulp mill service

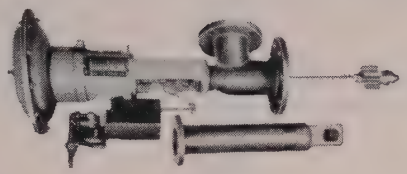
MIXERS



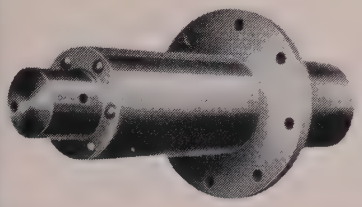
Dorr-Oliver Chlorine Mixer— rubber lined pressure chamber with internal counter-rotating propellers.

Dorr-Oliver Double Shaft Heater Mixer — rotating assemblies feature alternate propelling vanes and agitating pins. Reinforced stainless steel shell. Bearings outside shell for better protection.

NOZZLES



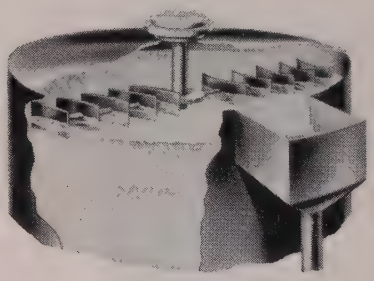
Dorr-Oliver Tower Dilution Nozzles — diaphragm actuated design features uniform, chatter-free throttling with freedom from plugging.

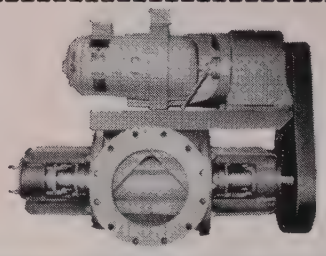


Dorr-Oliver High Density Storage Tower Nozzles — unique design with rotary oscillating action insures minimum wear and leakage.

MISCELLANEOUS BLEACH EQUIPMENT

Dorr-Oliver Top Scraper — rubber covered demountable rake arm provides continuous movement of stock towards launder. Adaptable for low density upflow bleach or chlorination towers.





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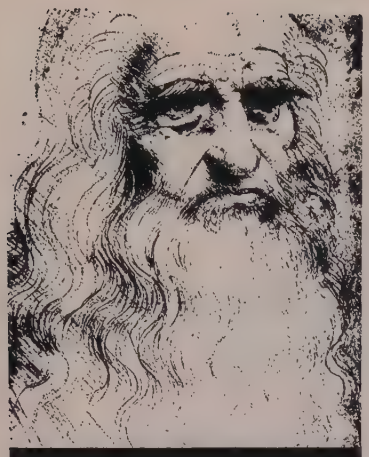

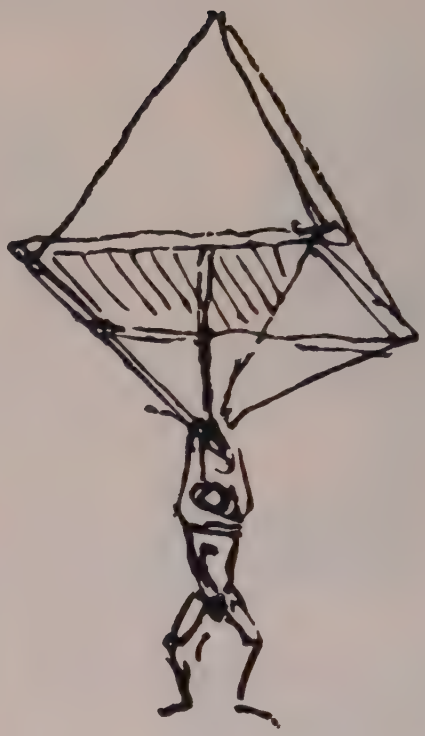

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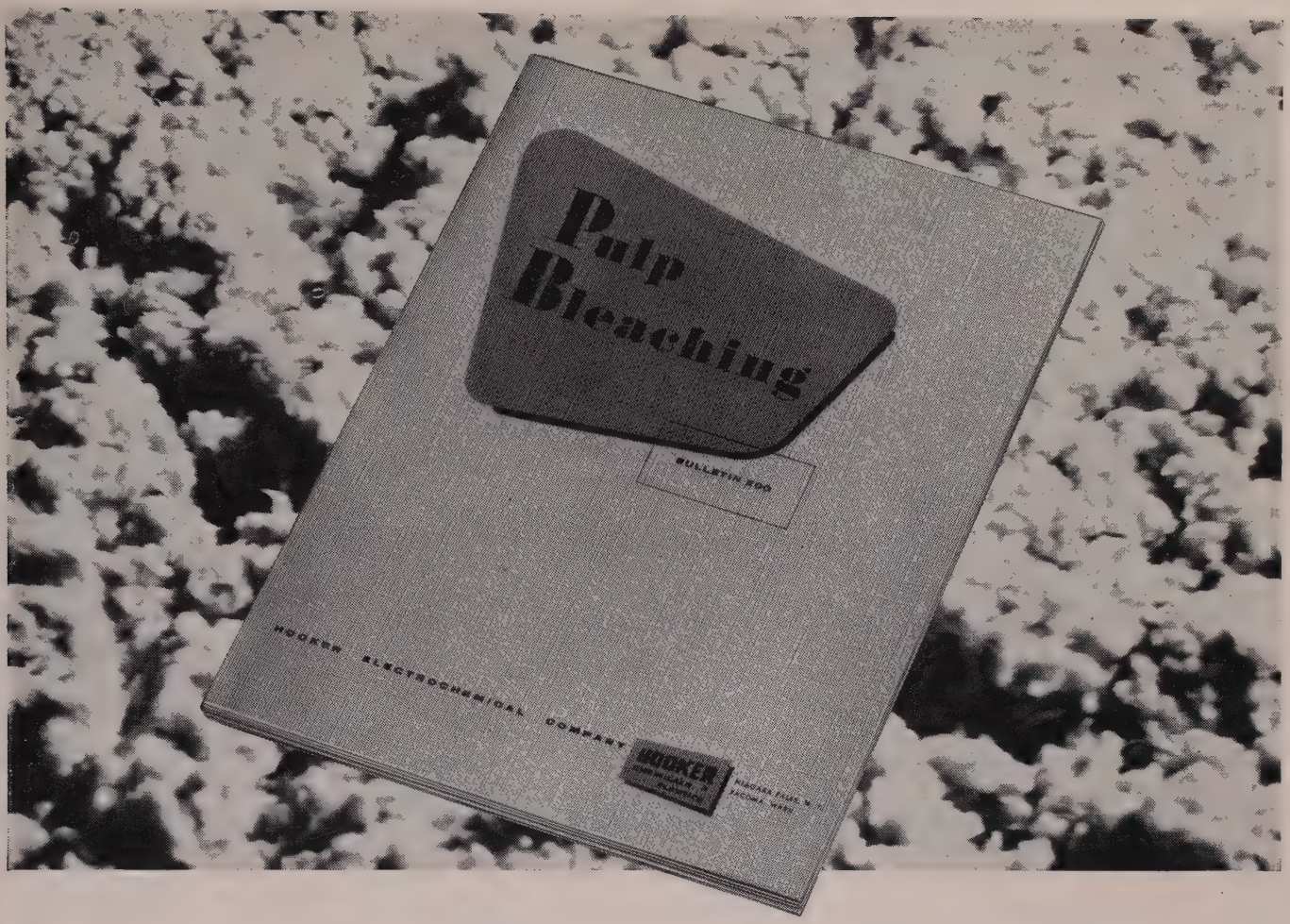

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The Mature Local Section

R. G. MACDONALD

Since the end of 1927 it has been the pleasure of your speaker to be interested in the establishment of the local sections of the Technical Association. After considerable maneuvering the first section was organized on the Pacific Coast. Like all sections the interest in forming the Pacific Section was self-generated. It was promoted by a few local men first as an independent body and eventually as an affiliate of the Technical Association. Considerable politics were involved and some corporate opposition was encountered. This opposition was based on the undesirability of permitting employees of competing companies to mingle and to exchange experiences. There was also the fear that the Association would eventually create standard procedures and publish information that would add to knowhow. These were justifiable fears since these things have been done. It is doubtful if many company executives would now have it otherwise.

Most of the groups that later became local sections started as independent pulp and paper clubs and had to demonstrate that they had the nucleus of a permanent body. This procedure had its drawbacks and advantages. The major drawback was the necessity of a few persons to shoulder the work and responsibility of interesting other people to join with them. Fortunately, all of these clubs had leaders who had the vitality and interest to do the job required of them.

In recent years the local groups found that they would require the support and backing of the management of the mills in their respective areas. The first eight or ten sections were organized without endeavoring to obtain such support and, although they have all survived, it would have been advantageous if they had attained such sponsorship, difficult as it was to do at the time. The first real effort to assure such a relationship was made by the parent of this group meeting—the TAPPI Southeastern Section. An excellent job was done by Glenn Kimble and his associates and an exceptionally strong organization was the result. Several years before another group organized in the same region without corporate sanction and lasted only a year or two. Another section recently chartered operated for a year or two without management endorsement and failed in its first petition for a charter. The deficiency was corrected and it is a good section.

The experience of this aforementioned group led one of our established sections to feel sympathetic to it and invited another group, then in formation, to become a district of it. Thus the local district as contrast to a pulp and paper club was born. The Association has districts in California, Indiana, Missouri, and New England which should eventually become sections. One section, Empire State, has districts but that is another story since its districts are, in a sense, five chapters making up the whole. They were formed as subdivisions during the gasoline rationing days and have remained as chapters ever since, getting together once each year as the Empire State Section in a fine three-day meeting up on Whiteface Mountain in the Adirondack State Park.

It is interesting to note how the sections have had their beginnings. One thing in common with all of them has been the existence of one or two individuals who wanted to have the benefit of such an organization. The first step has been a conversation, often casual, with the Association Secretary whose advice usually was to go ahead arrange the first meeting, select a place, arrange an *interesting* program, and invite a number of persons to be present. Assembling a group for such a purpose, and with the demonstration of an actual meeting was usually sufficient to set off the spark of enthusiastic desire for a permanent local group. The headquarters staff assisted in many ways and gave advice and guidance for proceeding without much fumbling. A major item of advice was the advisability of obtaining corporate approval. Such approval is now relatively easy to obtain but it must be solicited.

At the 1957 annual meeting of the Association two persons approached your speaker with the same mission, although having different geographical area interests. They were Ed Doyle of the St. Regis Paper Co. in Pensacola and Bob Fuller of the Gulf States Paper Co. in Tuscaloosa. Although Ed was thinking of the Gulf Coast region and Bob of the northern Alabama, Georgia, and Tennessee region, they evidently decided to combine their interests for the time being and to work together to establish a section west of Macon, the farthest west, of the Southeastern Section.

With the encouragement given by the Association headquarters, the group quickly established identification with the Southeastern Section and became one of its districts. The Section provided much assistance and held one of its meetings in Pensacola to show how such an event could be handled.

Ed apparently had no trouble obtaining interest on the part of others in the nearby region. He and his associates then sent out a call to all TAPPI men that they felt might be interested. It was then that I discovered the extent of the Gulf Coast. It started in Key West and continued to Mexico, and evidently included the rivers that drained into the Gulf, for invitations were sent to members in Louisiana, Texas, and Arkansas.

Although the Association has been rather deliberately lax in outlining section areas it had been trying to encourage the formation of another prospective section in the area covered by the states mentioned. The response to the cry of alarm was intended to be soothing, since the Gulf Coast group responded that it was already planning to be a parent of other groups that might be established in states along and near the Gulf.

R. G. Macdonald, Assistant to the President TAPPI, Gulf Coast Section Charter Presentation Address, New Orleans, La., May 15, 1959.

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So much for the background history of the Gulf Coast District. On an occasion such as this your speaker, who has had the privilege of speaking during the charter ceremonies of all of our sections since 1928, likes to offer a message based on his observations and experiences and which should be of value to all of the TAPPI Sections.

My message, at this time, is this—go out of your way to take a continued interest in your Section. Most of the sections have this hard-core of personal support. During the trials and tribulations of the early days of any district or section there is an almost crusading or evangelical fervor created. This carries over for a few years. The elders then often feel that they have done their job and someone else should "carry the ball." To assure continuity it is important for the elders to pass the reins to other individuals but it is then that the first faltering steps are noted.

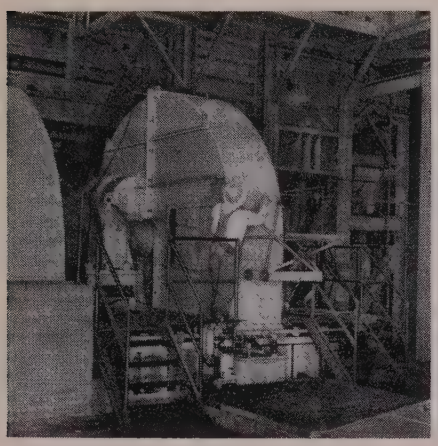
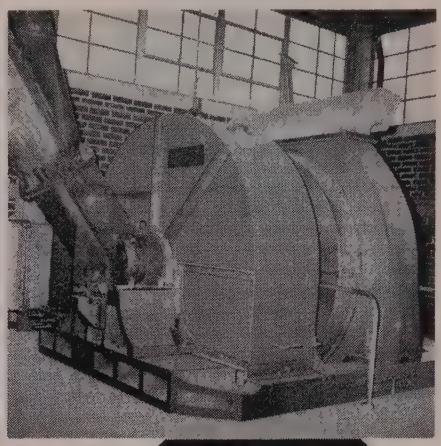
The new leaders often have not had the exhilerating interest common to the pioneers. Ways and means should be worked out to deal with the transition period which comes 4 or 5 years after a section is chartered. Our most successful sections have met this challenge and I am sure that the Gulf Coast Section will do well but some of our sections that have to contend with urban problems and a scarcity of a multiplicity of mill units find the going somewhat rough at times. No section has failed but several have faltered. In most cases the latter have been put back on the track by a few individuals who have had pride in their groups and considered the time and effort that they could give to be worth while and justified.

A corollary of that specific message is to take an interest in the National Association. This may seem to be a little more difficult to do since the assumption is that there is someone else interested in the national body who will attend to its affairs.

By taking an interest in the national affairs of the Association I mean to do so as a section. At the present time something is happening that can affect every member and every local section. Some sections have been alerted by their leaders and have taken such action as appeared to be feasible. I am referring to the ballot which has been sent to all voting members, and which provides for a change in the Association Constitution relative to the mechanism of amending it.

The section located geographically in the way that could be most affected—the Pacific Section—has protested the change and has asked other sections to vote against the proposed change. It has been interesting to note the early returns of ballots sent in by individuals. Where sections have alerted its members to the importance of the ballot many negative votes have resulted. Where members have had little, if any, direct advice, there has been a tendency to vote positive. The final total vote may favor the proposed amendment but it does illustrate the responsibility of all members to vote with considerable thought. If the amendment is approved, a sequence of amendments can follow rapidly changing many aspects of the Association as we have known it.

None of these future proposed amendments should be rubber-stamped and approved without the

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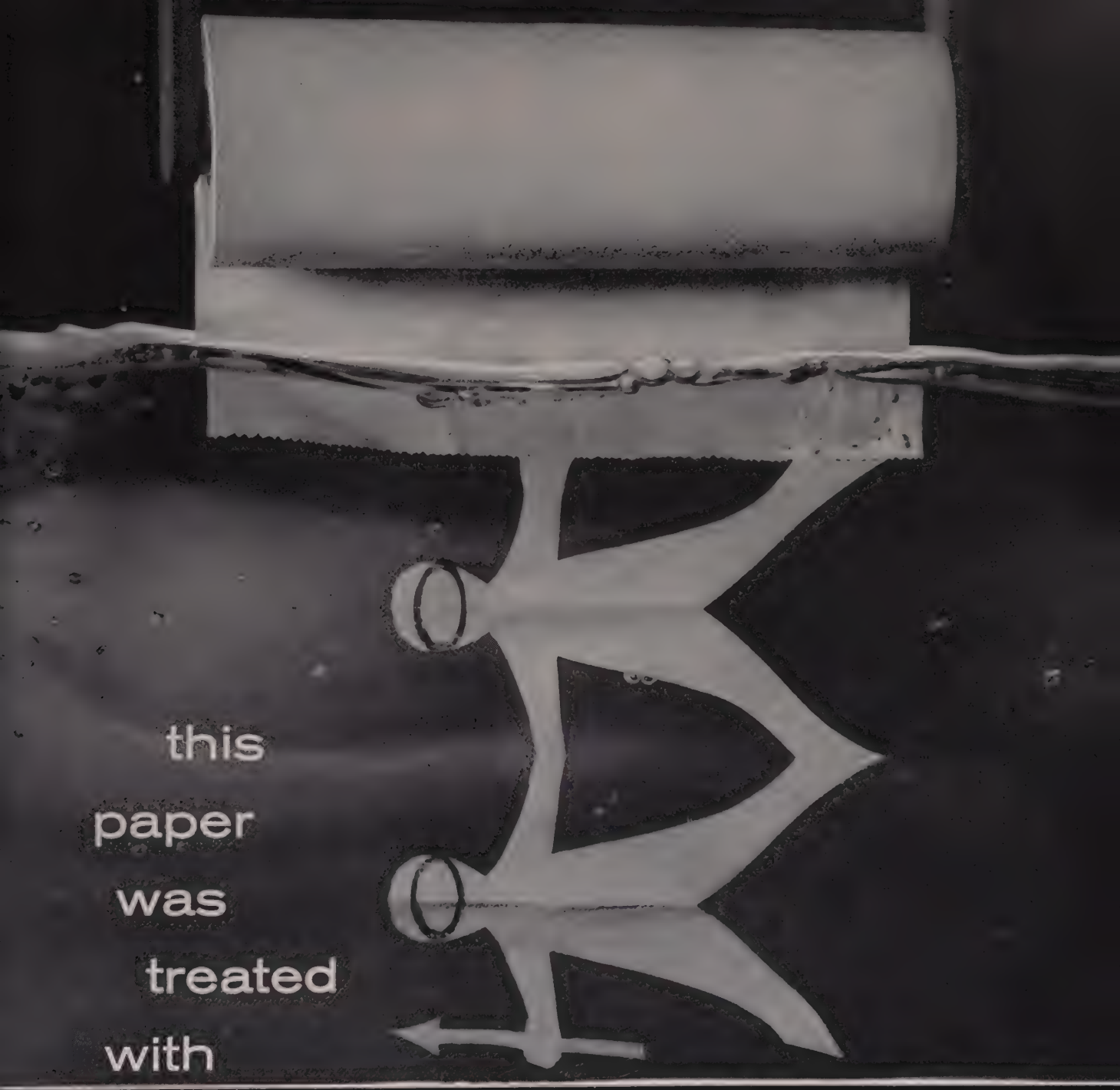

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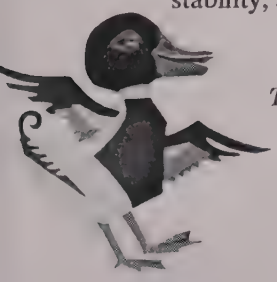

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combined thought of you as individuals and of your Section as a vital segment of the Association. For example, the Constitution and Bylaws mentions Sections only briefly and as we now know—somewhat vaguely.

Article XII of the Association Constitution provides for the establishment of local sections wherever there are *enough* interested members to form an adequate local section. The formation of a section can be authorized by the executive committee upon the written petition of *three or more* active members residing in the territory *embraced* by the proposed section. The section may receive annually for local use *such portion* of the annual dues of its members as may be *authorized* by the executive committee, said amount not to exceed 10% of the paid association membership dues for the current year.

No reference to sections is made in the Bylaws other than the reference to the local sections operations committee. Nothing is said about the definition of a section member other than to require that the section officers (also not defined) be members of the national association. These items will need to be spelled out in the near future. When they are spelled out you will be asked to cast your ballot. Will it make any difference which way the vote goes? Many members are interested only in the publications that are received from the Association. A high type of Association citizenship is needed. What other focal points exist for the crystallization of opinion that is more important than the local section?

The Bylaws vest much power in the executive committee. This is common practice and it is to be assumed that the executive committee can be trusted. It can increase your membership fee. It can decide what expenditures will be included in the annual budget. It can decide how much money can be given to a local section—from no money at all to a vast amount depending on what other financial objectives may be incorporated in the budget. It can allocate funds to finance research projects in educational institutions or other places. It can appropriate funds for new member services. All these things and many others can be voted by the executive committee, the members of which are elected by you and are therefore your representatives. Perhaps your only concern is your annual dues bill. At the time of its payment you may ask yourselves if you are getting your money's worth. The values received may satisfy you and besides you may conclude that there is nothing that you can do about it anyway, other than to pay up or resign. Occasionally a member will voice his resentment to certain things done by the Association and write a letter. A response and an explanation will be received and the letter may influence future policy.

A similar protest or comment presented to a local section might focus attention on a matter at the grass-roots level. A section has more influence than it may realize. It is represented on the Joint Advisory Council which has the right to meet with the Association President and have its opinions registered and referred to the executive committee for action.

Finally let me add one more word of advice. Have your section do things that will make you proud of it. This means electing officers and executive committee men who will meet as often as possible to discuss section affairs. It means having well attended meetings held as frequently as feasible and having a program that represents judicial planning. It means having reports of meetings published that say as much as possible beyond merely stating the subject and the speaker's name. (Comments or discussion of the section's business affairs and its attitude toward national TAPPI's actions can have considerable propaganda value.) The published report with pictures sets off your section with others. A sketchy report or no report at all can be symptomatic. Its effect on your members can be pride stimulus or disinterest. Always remember that the support of your next meeting depends to a measurable degree on the over-all success of its preceding meetings.

With this example of how to blow off steam for what I believe to be the common good of local sections I wish to welcome the Gulf Coast Section in behalf of the TAPPI Executive Committee as a duly authorized and full-fledged constituent part of the Technical Association of the Pulp and Paper Industry. The Association is proud of your progress to date and it hopes that you will do all the things that you can do to be increasingly proud of your important organization.

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Trends in Clay Coating of Paperboard

WILLIAM H. AIKEN

FIGURE 1 shows the daily capacity for producing clay coated board in the United States for the last 20 years. A look at this curve indicates the difficulty in trying to project future trends of coated board. An analysis of what happened in the coated board field until 5 or 6 years ago is relatively simple. The paperboard industry did not participate in the vast amount of work carried out by the paper industry on machine coating during the "inventive 30's." However, late in this period, some manufacturers of paperboard became interested in this development, and in 1939, the first modern machine coater was installed on a board machine. This was followed by another installation in 1943, and a third installation in 1945. No doubt this incubation period was greatly extended due to the war. Then, after the war, the industry rapidly installed coating capacity until 1948. It then sat back to digest what it had installed and for 5 years there appeared to be a small steady growth in coating capacity in the paperboard industry.

Those who would project the future on trends of the past would have said at the beginning of 1954 that coated board had fairly well stabilized and would enjoy a continued but steady growth in the future. However, even then, there were developments and trends which indicated that there would be nothing stable in the status of paperboard grades for years to come. Even after taking these into account, it is likely that in 1954 no one could have projected the recent tremendous increase in coating capacity in the board industry as indicated in Fig. 1.

Figure 2 shows that this growth in coated board capacity came from several different areas. The curve shown in Fig. 1 through 1953 is that for filled boxboard. Figure 2 shows that beginning in 1954, the coating capacity on filled boxboard showed a substantial increase in growth rate. Beginning in 1955, coated bleached board entered the market and has shown a spectacular growth since that time. For many years, there has been a small amount of coating of test liner board. Beginning in 1958, this capacity increased substantially, and it now appears that a trend is developing for more coating of this grade.

An analysis of the present status of coating in paperboard and what it is likely to be in the future is therefore quite complex and must take into account the general trends in paperboard use and manufacture. There is a paperboard machine in Middletown, Ohio, which can perhaps serve as a brief outline of what has happened and is happening to coated board, especially in regard to the filled sheet. On this machine is the first modern machine coater ever installed on a paperboard machine. It is a Consolidated letterpress print roll coater. In addition, this machine has a Champion scraper coater, a Warren air knife, and also the first vacuum forming cylinder ever successfully run on a paperboard machine. For many years, the letterpress print roll coater produced a quality second to none in the industry. Then, as quality demands increased, a Champion coater was added in order that the coating weight applied by the Consolidated coater might be reduced, which automatically cut down coating pattern and improved print quality. Eventually, an air knife coater was installed to improve quality and give better covering power for a given amount of coating. These adaptations of existing coating methods are typical of what has been going on in coating in the paperboard industry. Along with this has been the realization that even with the good base sheet which is possible through the use of a multi-ply sheet made on the cylinder machine, a better coated sheet quality could still be made by improvement in formation through such methods as a vacuum cylinder. Another development at this company indicates further the trends in the board industry in the future. This company is currently building a very complete coating pilot plant which incorporates the flexibility of an erector set and will allow a study of new coating processes, combinations of coating processes and drying methods.

WILLIAM H. AIKEN, Director of Research, Diamond Gardner Corp., Middletown, Ohio.

TRENDS IN BOARD USAGE

In order to understand the trends which have caused the very large increase in coating of paperboard, a look at trends in board usage is useful. The grade of paperboard most universally used for printing is referred to as folding boxboard. This is the grade normally made into the multitude of folding cartons used to package such items as those found in grocery stores and drug stores. Traditionally, these cartons have been made from filled boxboard which is made on a cylinder machine. This is a multiple sheet of five to nine plies. The two top plies are normally made from white fibers and the back of the sheet is normally made from reclaimed fibers such as old newspapers. Until quite recently, the only purpose for coating this sheet was to improve print quality. Now there is a trend toward coating methods which will not only improve quality but will give better hiding power and allow the use of fibers of lower brightness in the top liner.

With the introduction of the paper milk bottle, the solid bleached pulp sheet began to find substantial uses. Conventionally, this sheet is made on a fourdrinier machine at a site adjacent to a pulp mill. There are also a few producers of this grade using cylinder machines. As this sheet was tried for other uses, it was found to be inadequate for fine quality printing. Manufacturers of such sheets have resorted to the incorporation of hardwood fibers into their sheet and to coating to improve the print quality of the sheet.

In the last 5 years, there has been a large increase in the capacity of bleached board. At the same time,

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there has been a substantial increase of productive capacity of filled boxboard, primarily through machine improvements. This has resulted in a substantial overcapacity of paperboard for the last several years. There has been a vigorous sales campaign on the part of the manufacturers of bleached board to invade markets formerly held by producers of filled boxboard. In order to do this, the bleached board manufacturers have had to modify their processes, including installation of coating equipment, in order to make a sheet of print quality approaching that of filled boxboard. In order to meet this competition, the ingenuity of the producer of filled boxboard has been taxed to improve his quality and at the same time to reduce his costs to keep him in a competitive situation. This excess of boxboard capacity still exists and is likely to exist for some time to come. In the past several years, this has been the greatest stimulus for the development of coated boards and is likely to continue to be a very strong driving force. Both of these segments of the paperboard industry will certainly survive, but the split of the business in the future will depend on quality and economics.

In the meantime, other grades have entered the coated board field. The largest segment of paperboard production is liner board. The growth in this field has been in unbleached kraft board, most of which is made on fourdrinier machines. However, there is still a substantial and fairly stable portion of liner board made from jute. This grade is made on cylinder machines from reclaimed wastepaper. In recent years, there has been a tendency to upgrade the corrugated container. This has taken the form of bleached kraft liner board for the outside ply, white fibers on a secondary headbox, and coating of unbleached board. For many years, there has been a small production of coated unbleached board for corrugated containers, but this has significantly increased in recent years. Unbleached kraft liner board has also been used for folding cartons. The first appearance was in bottle carriers and a white surface was added by a printing process. More recently, there has been introduced into the carton business liner board coated with an air knife coater, both on and off machine. Because of the nature of the base sheet, to date this grade is competitive only in fields with reasonably simple printing. However, coated unbleached kraft and jute boards must be considered in the future picture of coated paperboard.

DIFFERENCES IN PAPER AND PAPERBOARD IMPORTANT TO COATING

In order to understand the trends developing in coating of paperboard, it must be realized that there are many characteristics of paperboard which are important to the coating process which are different from paper.

Interest in One Surface. In most of the uses of coated board, there is interest in only one surface from a printing standpoint. Therefore, most coated board is coated only on one side. There is, however, a small amount of board coated two sides from an appearance standpoint.

Coating Is a Relatively Small Percentage of the Weight of the Sheet. Board is thick compared to paper, and therefore, coating is a relatively small percentage of the weight of the sheet. Board will, therefore, absorb substantially greater quantities of water (applied either as heavy coating weight or as a lower solids coating) than will paper. This makes it possible to use a variety of coating methods on the board machine.

Board Is Produced at Relatively Slow Speeds. Because of the necessity of drying the heavier sheet of board, board machines usually run slower than paper machines. This also simplifies the use of coating processes on the machine.

Board Is Sold by Caliper Designation. Although board is sold both by weight and sheet count methods, it is sold to a rigid caliper specification. For most uses for paperboard, stiffness is the most important physical property and stiffness is highly dependent on caliper. The user of board, of course, uses it by the square foot, so he is interested in the lightest board possible for a given caliper consistent with good print quality. However, full caliper is expected. Therefore, with other things being equal, application of coating adds to cost of the board on a yardage basis.

Board Is Usually not Supercalendered. Although at the present time, gloss is a desirable characteristic in coated boards, only a small percentage of the board production is supercalendered. This is due both to economical and quality reasons. First of all, the supercalendering operation would reduce the caliper substantially, which would necessitate producing a thicker and heavier sheet to begin with. Supercalendering also tends to destroy dry hiding of the coating, and since even the best of boards are somewhat nonuniform in formation, supercalendering tends to give a mottled effect.

Waterproof Coating. In general, coated boards must be waterproof. A coated suit box that would rub off on a lady's dress if she were caught in a shower or coating on a soap box which would come off when contacted with wet hands could be disastrous. Therefore, a very large percentage of coated board production has excellent waterproofness. This is one of the primary reasons why casein and soybean protein binders, either with or without polymer emulsions, are used almost universally in paperboard coating. Waterproofing is achieved by incorporation of insolubilizing agents in the coating itself or by their application at a water box on the calender stack.

Gloss Ink Printing. A substantial portion of coated paperboard is printed with gloss inks. These inks are prepared from heavy bodied varnishes which tend to stay on the surface of the board to give a high gloss finish. However, such inks are very tacky, and therefore, demand a coating with high pick resistance. Since it is desired that the inks stay on the surface, the coating must be relatively nonabsorptive. Clay or a combination of clay and titanium dioxide are almost universally used as the pigments for coating boxboard.

Scoring and Folding. Board for folding carton uses must not crack when scored and folded into a carton. The coating, therefore, must have sufficient flexibility to withstand this treatment.

Scheduling Problems. Filled boxboard is made to order. Although there are some substantial runs, there are many short runs and a week's production schedule may include coated board for letterpress gloss ink printing, letterpress flat ink printing, offset printing,

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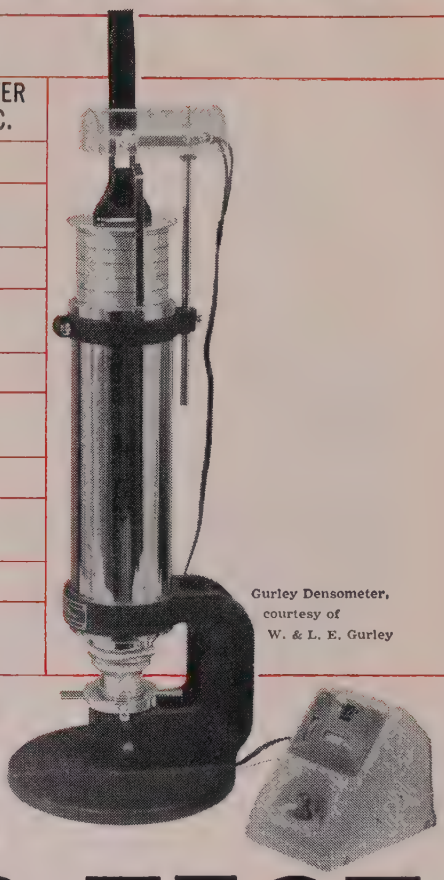

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and gravure printing. Many times the size of order will require less than a full batch of coating. This problem has been solved by development of a universal type coating which will perform satisfactorily for all printing processes. Sometimes the gloss ink characteristics are modified by calender treatment with water-soluble gums either before or after coating.

Trim Problem. Trim is an economic factor in the production of all board, but more so in the production of filled boxboard, because the trim must be downgraded to filler stock value. Therefore, the typical boxboard producer will run a variety of grades on each of his machines during a given week in order to get the best trim possible. This means that on a filled boxboard machine, the coating equipment will not be used a considerable amount of the time. For example, the entire production of coated filled boxboard made in 1958 could have been produced in 55 running days if all of the coating equipment available had been utilized.

IMPORTANT FACTORS IN COATED BOARD QUALITY

There are a number of factors important to the production of a quality sheet of coated board. While some of these are fairly well known, many are still not completely understood.

Base Sheet

The most important factor in coated board quality is the base sheet. Failure to recognize this has led to many of the difficulties encountered in producing a quality sheet of coated board. There are those who have gone on the assumption that painting a poor piece of board would make it good. The painting may certainly improve the whiteness and may even improve ink holdout, but something more is required to make a sheet with good half-tone print quality.

Furnish. Furnish is extremely important in the production of a quality sheet of coated board. The flexibility of furnish on the cylinder machine makes it easier to produce a good printing sheet by this method than is possible on the fourdrinier machine. Most producers of fourdrinier bleached board have learned the hard way that a sheet of 100% bleached kraft softwood fibers may give a very strong sheet, but such sheets are very difficult to print. Many of these mills are now incorporating substantial amounts of hardwood fibers into their sheets. The best printing fourdrinier bleached sheets might be described as thick sheets of book paper.

Formation. Formation is of great importance in the printing quality of coated boards. It is a quality factor in that uneven formation will cause a mottled appearance, especially on wet finishing. Formation deteriorates as speed increases, so efforts to get greater productive capacity have been counter to good quality coated board. This is one of the reasons for the tremendous amount of work going on at the present time on the design of paper forming equipment. Some of the newer forming devices which give better formation and are run at higher speeds will certainly result in better coated boards and/or reductions in cost. These developments in forming equipment are areas to watch for trends in the future of coated board.

Unbleached kraft boards are used primarily in applications where their great strength is required. Both the furnish and formation of unbleached kraft boards are undesirable from a printing quality standpoint as a base sheet for coating. To date, coated unbleached boards have been used in applications which require only simple line printing. Broader use of these boards for higher print quality will depend on improvements of the board through the use of a secondary headbox or the development of coating methods which are more independent of base stock quality.

Smoothness. Smoothness of the sheet before coating is of utmost importance. In the case of the Champion coater, which applies coating to the wet sheet, this smoothness must be achieved in the forming and pressing section of the board machine. With processes which use a dry sheet as base, some of the smoothness can be imparted in a breaker stack.

Wire Side versus Felt Side. Practically all filled boxboard is coated on the wire side because this is the side which is normally white lined. The question of whether fourdrinier board should be coated on the wire side or the felt side seems to be one of the questions which is still not completely resolved.

The "Live" Sheet. Many people feel that there is a great advantage to coating the sheet on the board machine while it is still in the "live" or plastic condition and can be easily formed. Advocates of this belief contend that this makes it possible to produce a better coated board on the machine than can be produced in a converting operation off the machine. While this argument has not been conclusively resolved, there is no doubt that the best machine coated boards and the best off machine coated boards are quite similar in printing quality.

Coating Formulation

Coating formulation is roughly standard for coated board. A typical formulation would contain 15% binder and 85% pigment. The binder typically would be casein or soybean protein. Part of the binder might be a polymer emulsion to improve printability and bender, however, in some cases at the expense of light resistance and opacity. Pigment composition would be typically built around a high white domestic clay. Up to 25% of the pigment may be titanium dioxide.

Application Method

Four principal methods have been used for paperboard coating.

Roll Coaters. Both letterpress print roll and gravure print roll coaters have been used to coat board. These coaters are characterized by their ability to apply high solids coating and simplicity of operation. Unless very light coating weights are applied, an excessive film split pattern results which gives poor gloss upon varnishing and interferes with fine half-tone reproduction. There are still several board machines on which roll coaters are installed, but many of these are used to a limited extent. Off-machine reverse roll coaters are being used to an increasing extent for coating unbleached kraft and jute boards.

Champion Coater. In the Champion coater, an excess of coating of about 45% solids is applied by roll to the wet sheet in the drier section. A rotating doctor bar located several inches from the applicator roll scrapes off the excess of coating. Coating weight is determined both by coating solids and by the distance

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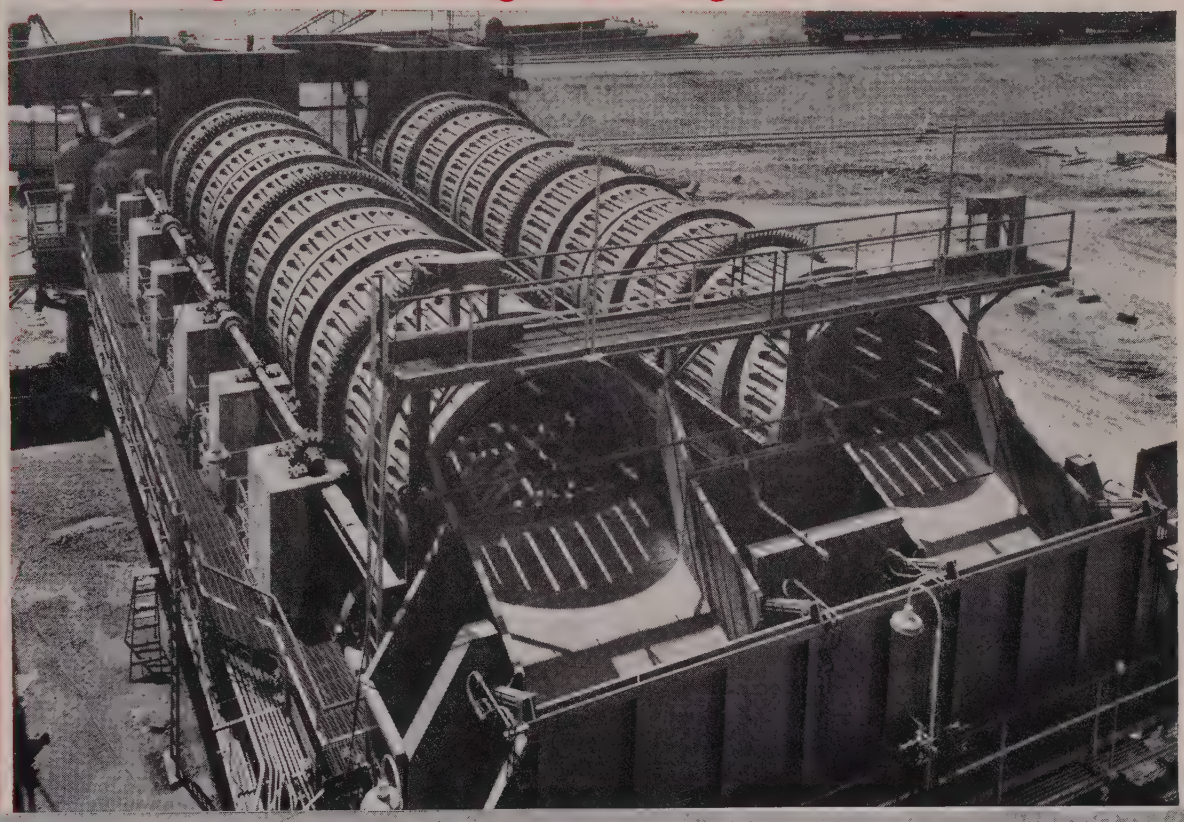

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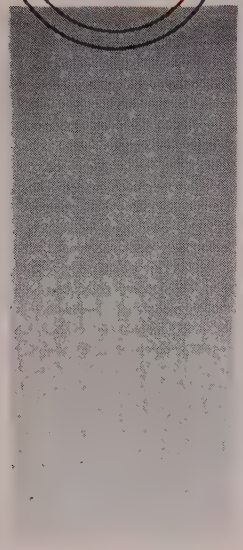

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between the applicator roll and doctor bar. Since the doctor bar tends to level the sheet, more coating will be left in low areas than in high areas. Unless the brightness of the base sheet is similar to that of the finished coated sheet, a mottled appearance will result. The Champion process is one of the better established methods for coating filled board, and it continues to grow in use. The print quality of board coated on the Champion coater is highly dependent on the quality of base sheet produced on the wet end. Some of the finest and also some of the poorest coated boxboards are produced with Champion coaters.

Air Knife Coater. In the air knife coater, an excess of coating is applied with an applicator roll and then some of the coating blown off with a stream of air. This coating method applies a coating of uniform thickness which follows the contours of the sheet. The smoothness of the finished sheet is, therefore, very dependent upon the smoothness of the base stock. The air knife coater gives the greatest hiding power for a given coating weight of all the methods commonly used for coating paperboard. It is being used in increasing amounts for coating darker body stocks; however, it is also popular for coating bleached board.

Trailing Blade Coater. The trailing blade coater has had the most spectacular growth of all coating processes in the coating of board. With one exception, all installations have been used on bleached board. Since this coater has a levelling action, more coating is applied to the low spots than to the high spots. Therefore, the body stock should be similar in brightness to the brightness of the coated sheet to prevent mottle. Streaking has been the major difficulty to overcome with the trailing blade coater.

At the present time, no coating method has been demonstrated to be superior to all others for producing a coated boxboard sheet of good print quality. Of the seven best printing sheets of boxboard made today, three are filled sheets made with Champion coaters, two are bleached sheets made with trailing blade coaters, one is a filled sheet made with an off machine air knife coater and one is a filled sheet made with an on machine air knife coater. While there are minor differences in the print quality of these seven sheets, all have excellent printing characteristics by letterpress, offset and gravure, and there is little to choose from between them. On the other hand, some of the poorest printing sheets made today are coated by trailing blade, Champion, and air knife. Apparently, there are other factors, primarily those of the base sheet, which are more important than application method.

Calendering

Calendering is important to impart smoothness and gloss to the sheet. Calendering is undesirable in that it increases density and reduces dry hide which tends to cause mottling.

PROBLEMS WITH COATED BOARDS

Unless proper care is taken, a number of problems may be encountered due to the use of coated boards.

Rub Resistance. Many users of folding cartons have very stringent requirements in regard to rub resistance of printing on the carton. These requirements are

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necessary because of rubbing which may occur as cartons vibrate in a shipping container during shipment. Rubbing on unvarnished cartons is more of a problem on coated board than on uncoated board, because the coating is apparently more abrasive and it is more difficult to obtain good ink adhesion on coated board. Rub is especially bad where the coated unprinted area comes in contact with a printed area. Care, therefore, must be taken in formulating inks for coated boards.

Gluing. Several types of gluing problems may arise due to the use of coated board. Gluing characteristics are usually specified by designation of some type of sizing test of the top and back liners. A specification for sizing for top and back liners which is satisfactory for a given packaging machine for uncoated board will not necessarily insure that a coated board meeting the same specifications will run on that machine. This is probably an inadequacy of test method to characterize the property important to gluing. In general, there have been no great problems in gluing waterproof coated boards once the proper specifications have been established. However, when a glue machine is switched from uncoated board to coated board, some adjustments may have to be made. Coatings not adequately waterproof may give poor glue seams, especially in humid weather.

Marking. Coated boards may tend to mark or take on a shine when rubbed in forming or gluing machines.

Bender. Bender can become a problem with coated boards. One of the best methods of insurance against such failure is the use of a base stock with adequate bender. If coating weights in excess of three to four pounds per thousand square feet are to be applied, part of the protein binder must be replaced by some flexible-type binder or else a brittle coating will result.

Abrasion. Coated boards are more abrasive than are uncoated boards. This shows up in increased wear of knives on cutting presses. Some people also feel that coated board causes excessive wear of printing plates.

Picking. Coated boards are more subject to picking than are uncoated boards. Many people using Champion coaters have found it necessary to take care to properly refine top and inner stocks to avoid liner picking. Boards to which heavy amounts of coating have been applied tend to produce an excessive amount of slitter dust; this is especially true if an all protein binder is used. Unless means are provided for removing this slitter dust, it may cause major dirt problems on the printing press, especially in lithography.

BOARD COATING EQUIPMENT

A survey has been made of the capacity available for coating paperboard in the United States. Summaries of this capacity are given in Figs. 1 and 2. This information was obtained by sending a questionnaire to the various producers of coated board with the request that they give the type of coating equipment in their plants, when it was installed, and the capacity of the machine. It is believed that this survey is fairly complete in regard to machine coating installations which were in operation by the end of 1958. There may have been some previous installations which have since been removed from the machines which do not reflect in these data. The capacity figures given are

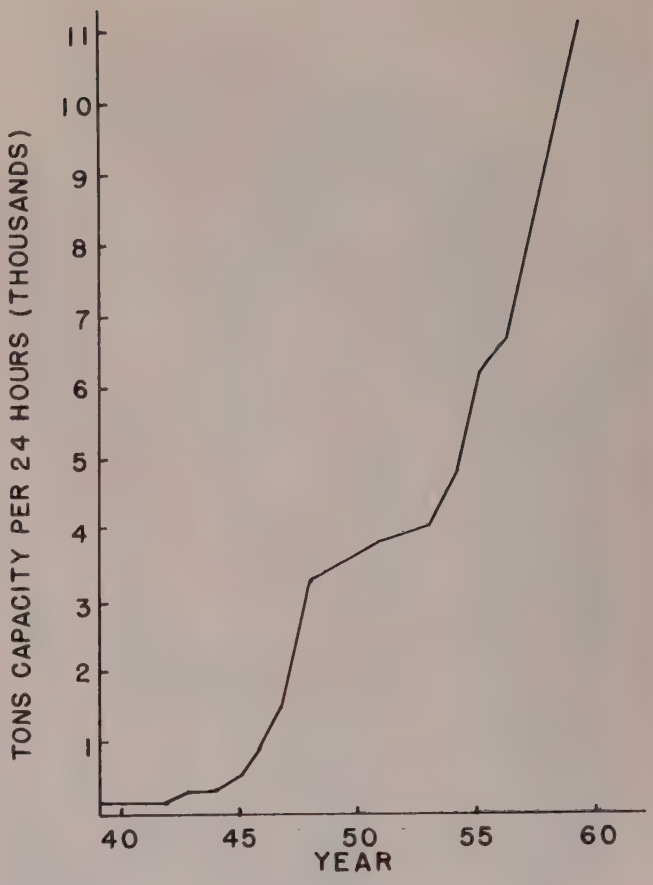


Fig. 1. Total board coating capacity

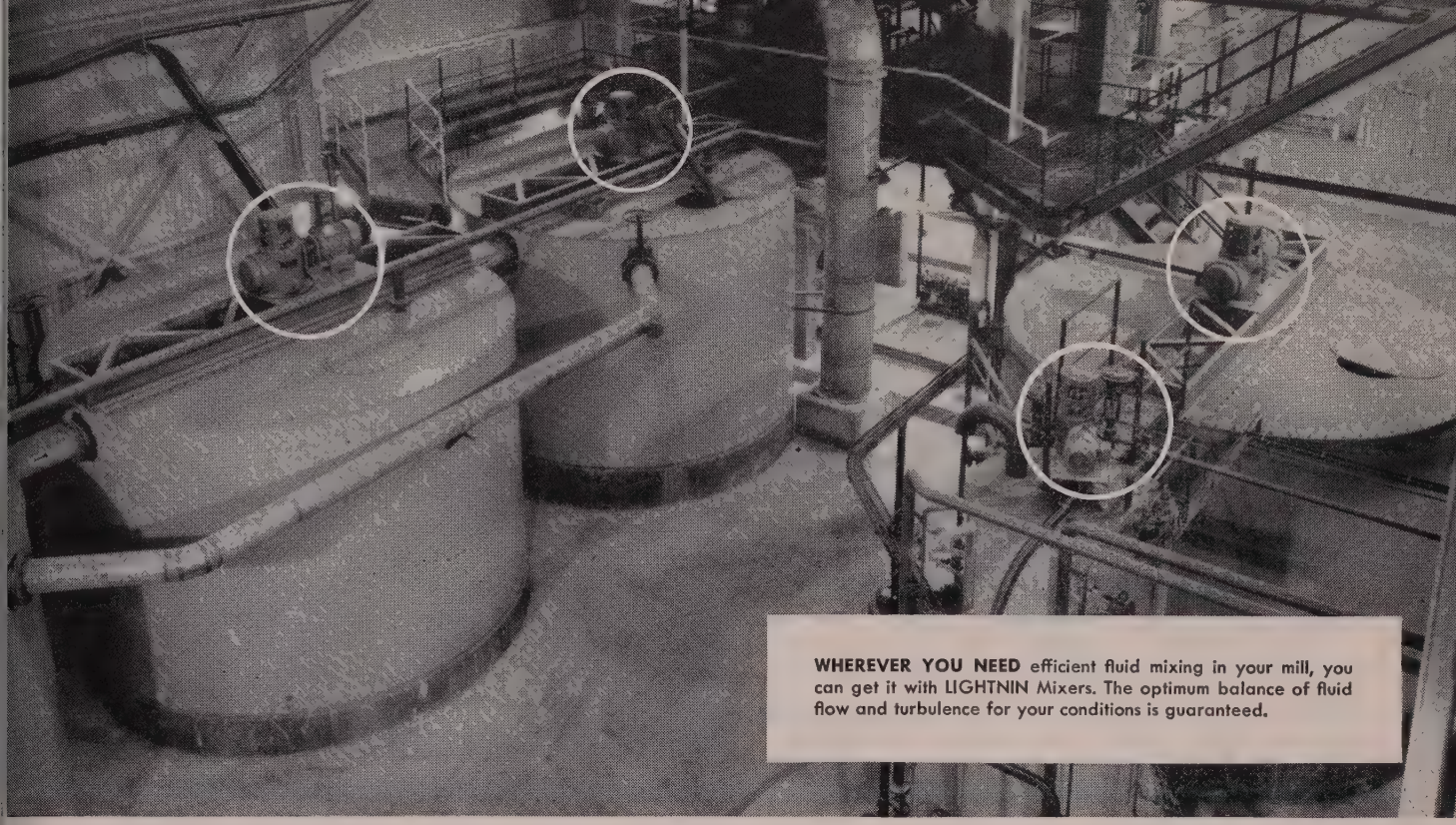
the current capacities of the machine and may, therefore, be somewhat different than when the coaters were installed. In those cases where several different types of coaters are installed on a given machine, capacity for each of them is shown, even though some of the units may no longer be in use.

It is known that the data are not complete for several installations which will be made in 1959. The data are also somewhat incomplete in regard to a few off-machine installations; however, these units are of relatively minor volume. It is believed that the data give a fairly accurate indication of trends in coating equipment capacity.

Figures 3, 4, and 5 show the capacity for coated board by type of process. It is seen that the capacity for roll coaters is relatively stable. As was pointed out earlier, much of this capacity is currently not in use. The Champion coating process has grown fairly steadily since the first unit was installed in 1943. All of this capacity is on filled boxboard. The air knife coater has been popular in recent years for both filled boxboard and bleached board. Recently, air knife units have been installed to coat unbleached board. The trailing blade coater has had a spectacular growth for use in bleached board. Below is given a summary of the coating capacity currently installed as shown by this survey.

Filled Boxboard

Twenty-two companies in 33 plants have 98 coating units on 45 machines for an average daily capacity of 118 tons per machine. The breakdown of the 98 coating units is as follows: 80 Champion units, nine



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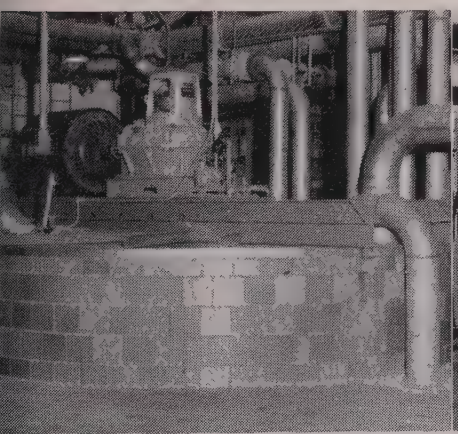

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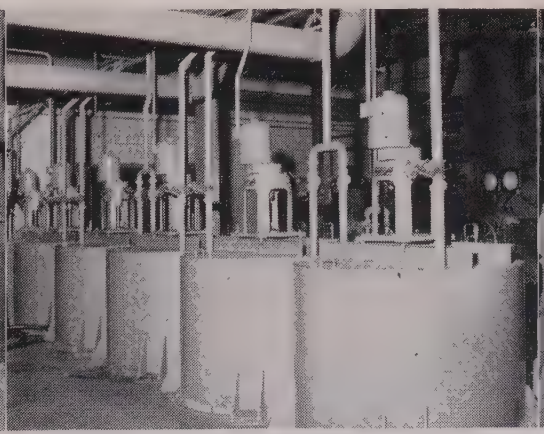

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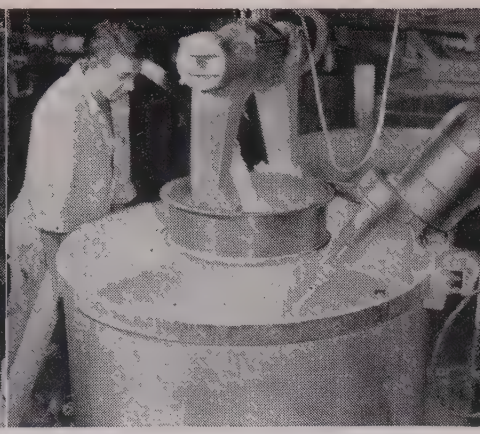

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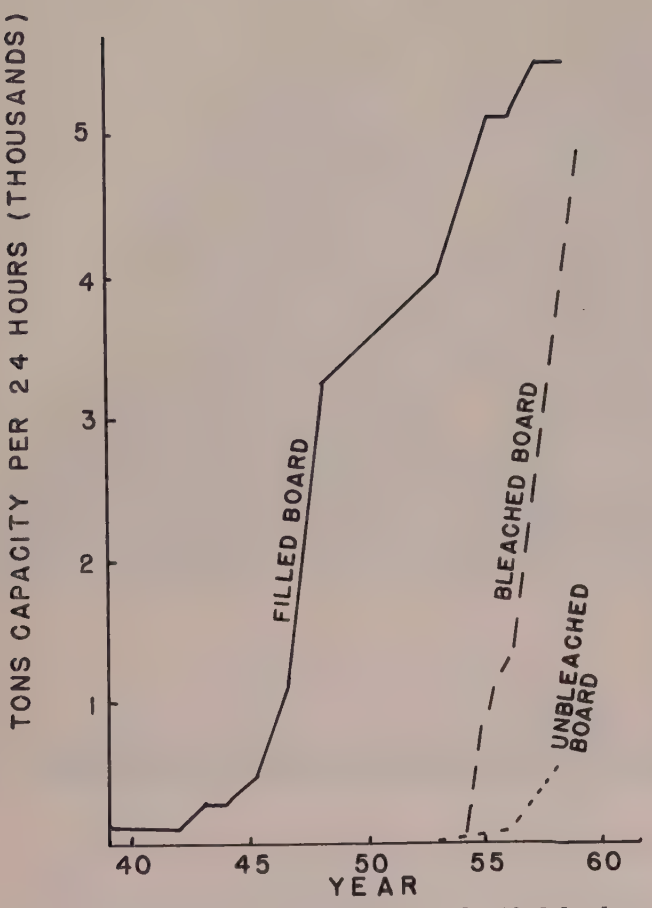


Fig. 2. Total board coating capacity classified by base stock

air knives, six letterpress print roll coaters, two gravure print roll coaters, and one trailing blade coater.

Three of the companies who have machine coating installations and two other companies have nine coating units on nine off-machine coating installations for an average daily capacity of 49 tons per installation. Eight of these units are air knife and one is a roll coater.

Bleached Board

Twelve companies in 14 plants have 25 coating units on 18 machines for an average daily capacity of 290 tons per machine. The breakdown of the coating units is as follows: seven air knives, two gravure print roll coaters, and 16 trailing blade coaters.

Three of these companies, who have on-machine coaters, also have six coating units on three off-machine coating installations for an average daily capacity of 133 tons per installation. Two of the coating units are air knives and four are trailing blade coaters.

Unbleached Kraft and Jute Board

One plant with an air knife on a machine has a daily output of 200 tons. Three other companies in four locations have five coating units on five off-machine installations, producing an average daily capacity of 67 tons per installation. Two of these coating units are reverse roll coaters and two are air knives.

Our survey shows that on a 6-day week basis, there is installed coating capacity to produce about 1,800,000 tons of coated filled board per year. This is about 73% of the total amount of filled boxboard produced in 1958. Actually only about 12% of the filled boxboard produced in 1958 was coated, indicating that on the average the coating capacity for filled boxboard was used about 17% of the running time.

Our survey indicates that by the end of 1959, there will be capacity installed to coat 1,510,000 tons of bleached board. This is about 98% of the bleached board produced in 1958. We have no figures available on how much coated bleached board was actually produced. However, in general, bleached board to be printed in other than very simple copy is coated.

TRENDS IN BOXBOARD USAGE

There are a number of trends in the usage of boxboard which must be considered before a look can be taken into the future. Some of these trends are counter to each other, but all must be taken into consideration.

Overcapacity. There is currently an overcapacity of facilities for bleached and filled boxboard. Despite this, plans are under way for expansion of bleached board facilities, and many producers of filled boxboard have improvement programs to speed up their machines in order to reduce cost. This overcapacity, which is likely to exist for several years, will continue to be a spur to both producers of bleached and filled boxboard to improve their quality and to lower their costs.

Demand for Gloss. At the current time, there is a substantial demand for a glossy coated board. This may continue or may be a passing fad. As gloss is increased, more coating weight is required to get a given amount of hiding power and this leads to increased costs. Competing with this trend toward a glossier board are ways to produce a high gloss printed carton by printing methods. In the long run, this will become a matter of economics and appearance.

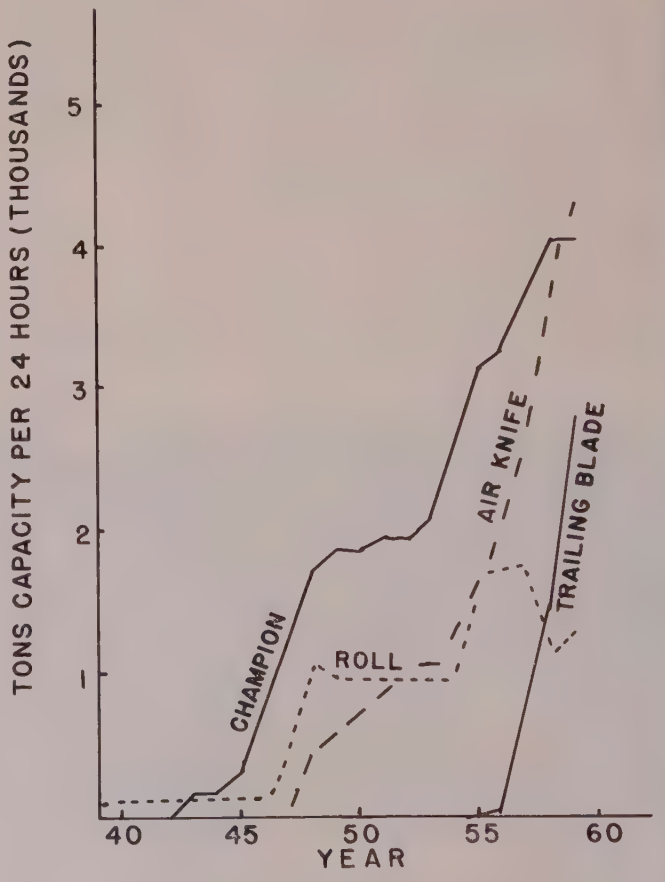
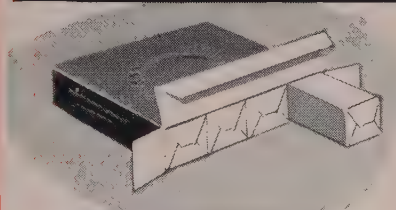
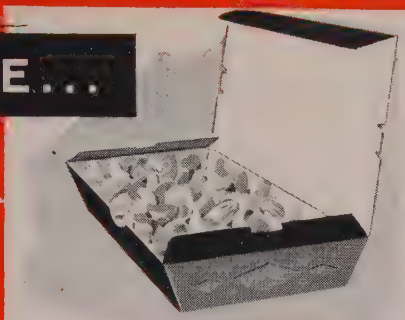


Fig. 3. Total board coating capacity classified by coating process

IF YOU PACKAGE...



BUTTER, OLEO



NUT MEATS

MILK, JUICE

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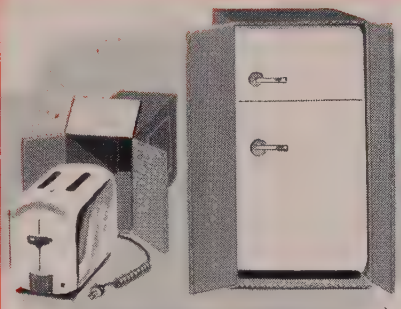
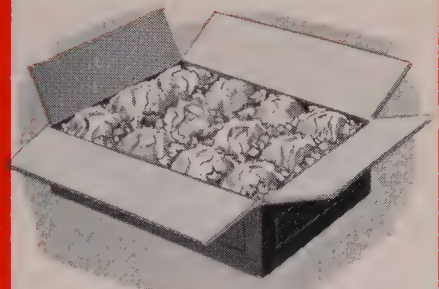


APPLIANCES

MEATS



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Upgrade in Print Quality. In recent years, there has been a very strong upgrading of printing quality with more and more printed board going to high quality half-tone printing. This has been largely accomplished by a very large upgrade in the printing quality of board, and this has been one of the factors which has led to improvement in coated board quality. On the other hand, there is also a trend toward the use of lithography in printing cartons. Lithography will do a better reproduction job on a rough board than will the other printing processes.

White Ink Printing. For many years, there has been a certain amount of white ink printing on both unbleached kraft board and filled boxboard of low brightness to give a white printed sheet. The use of this method compared to coating becomes a matter of economics and quality.

Captive Carton Plants. There is a very strong tendency for producers of board to acquire carton plants. The number of independent carton plants who are substantial users of coated board are continuously diminishing. This reduces the market for board as such and increases the competition for the remaining customers. This tends toward increased improvements in quality. On the other hand, it is generally true that a captive carton plant may be less fussy in regard to certain characteristics than the outside market. This tends to set the quality of board production to that which is adequate rather than at meeting every characteristic that every other producer comes up with.

FUTURE TRENDS IN COATED BOARD

Coating developments are in a very active state. To try to predict accurately the exact course that these developments will go could not be done without a crystal ball, which I do not possess. However, there are a number of trends which are evident and a number of areas in which work is very active.

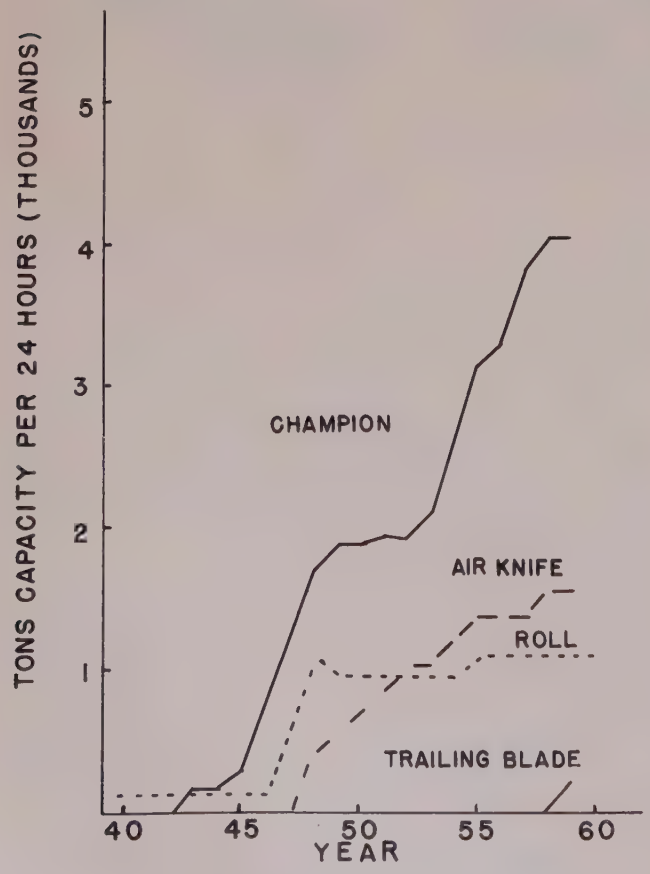


Fig. 4. Filled board coating capacity classified by coating process

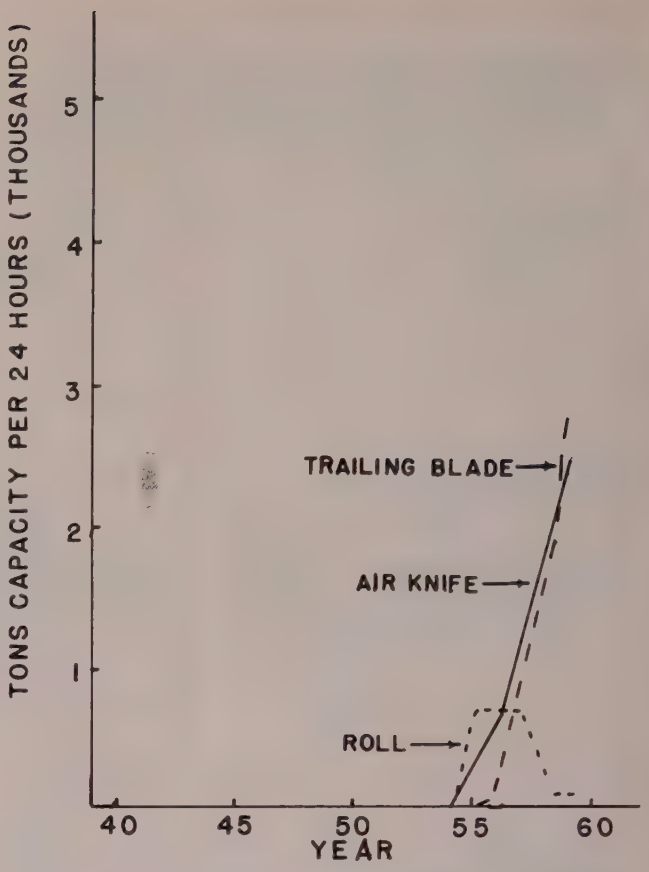
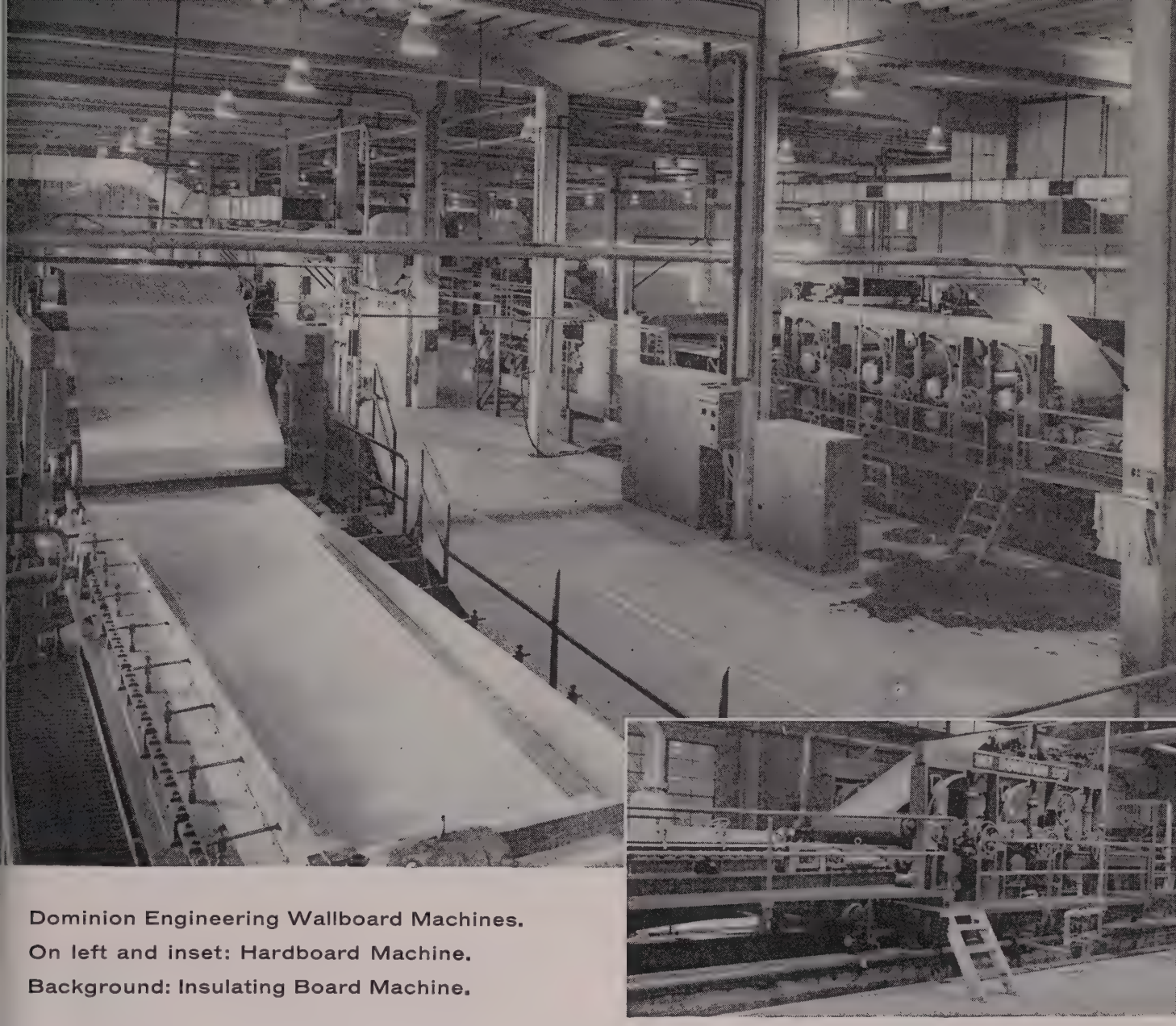


Fig. 5. Bleached board coating capacity classified by coating process

Bleached Board. The bleached board industry has grown very rapidly in the last decade and has made great strides in quality improvement. It has not only taken most of the uses for wet food packaging, where it originally got its start, but it has moved into areas formerly serviced by filled boxboard and unbleached liner board. In order to do this, many of the producers of this board have found it necessary to go to a mixed furnish and to take more care in the formation of their sheet. They have also found it necessary to install coating equipment. A few of these producers make a very excellent printing sheet, while others are marginal. For the marginal producers to continue in the highly competitive market, they must either find markets that do not require a good print quality, or they, too, must make a better base sheet and do a better coating job.

Many producers of the bleached sheet have found that coating a poor base sheet results in a poor coated sheet. In many mills the fiber economy is such that a mixed furnish is difficult to use. Considerable work is, therefore, being done on coating formulation and coating methods. There are still big questions to be answered as to which side of the sheet should be coated and the coating method to be used. Considerable work is being done on a combination of air knife and trailing blade coaters. Somehow the Champion coater seems to have been overlooked by the bleached board producers, but it, too, must be considered. For the pro-



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ducer of a good base sheet, the Champion coater or the trailing blade coater would seem the most economical way to produce a quality sheet. There is still work to be done to arrive at a coating method to produce a quality sheet from a poor piece of board, and no doubt work will be done in this area.

The continued growth of bleached board depends upon both economic and utility factors. Passage of the Kennedy bill to modify the Fair Labor Standard Act could cause a substantial increase in the cost of producing bleached board in the south.

Filled Boxboard. The increased capacity of bleached board has created an excess capacity of all types of board, and this, in turn, has affected the markets for filled boxboard. The producer of filled boxboard, in general, has a cheaper source of raw material than the producer of bleached board, but he must do a more extensive cleaning job to make it suitable for many end uses. In addition, filled boxboard is made on cylinder machines which run at relatively slow production rates. In an effort to produce a better sheet at a lower price, very substantial amounts of work are being done to improve the cylinder machine, as well as stock preparation processes. A great amount of work is being done on forming units to make the filled sheet. Out of this should come methods for making a clean, uniform sheet from wastepaper at a very substantial reduction in cost. In addition to this, the sheet will be a more suitable base for coating, which will inherently give this type sheet a further advantage as a coating base.

Up until quite recently, coating was used on filled boxboard only to improve print quality. This coating was applied to a sheet that was already lined with white fibers. In effect, the gray sheet was first veneered with white fiber and then painted with white paint. In recent years, there has been a concept in the filled boxboard industry that the veneer of white fibers, which is an expensive part of the sheet, should be eliminated and the gray sheet should be merely painted. The coating process used today which will do this is the air knife, and there should be a great increase in the number of air knife installations in the filled boxboard industry. The air knife is of value because it gives a uniform coating thickness which gives good hiding power. Experimental work will be accelerated on other methods which will give a uniform coating weight. Such methods as spray coating and extrusion coating, which have been largely abandoned in the past, will be looked at again. A machine which extrudes a curtain of coating and which has been used in the lumber industry has been recently adapted to the coating or corrugated board. This type of application will certainly be considered for coating of filled boxboard. The incentive for this type work by the producer of filled boxboard is the difference in cost of changing the appearance of a wall by applying paneling or by painting. There are certainly technical problems to be overcome such as color control, and when applying heavy coatings, bender and smoothness. However, these are not insurmountable technically, and if they can be solved, the dollar saving is sufficiently great to insure the manufacturer of filled boxboard that he can gain position in the competitive market.

The immediate major changes in coated filled boxboard will be a shift to air knife or other methods which allow the use of a lower brightness base stock. This shift will be made first in the higher brightness grades. Lower brightness coated grades and uncoated grades will shift to air knife coated sheets if this becomes more economically attractive.

Unbleached Kraft and Jute Boards. Increasing amounts of these boards will be coated to upgrade the appearance of corrugated containers. Beyond a certain point, print quality will be limited by the nature of the base sheet. This will be improved in certain cases by the use of secondary headboxes, but work must be done beyond this to get coating methods that will give good printing quality on this base sheet. Otherwise, high quality printing on corrugated boxes will require the use of bleached board. Coated unbleached liner board will continue to be used in carton applications requiring high strength, but these, too, will be limited if print quality is not improved.

Overall. During the 1930's, the paper industry did an excellent job of pioneering many types of coating methods. Then, there seemed to be a dormant period for 15 to 20 years while these methods were applied and adapted. More recently the imagination of equipment suppliers, paper producers, and board producers has been stirred by the concept of the trailing blade coater. This and other concepts must continue to be explored. The greatest need for coating equipment today is a method or a combination of methods that will make possible production of a high quality coated board on a rough base sheet. Whether or not this is possible remains to be seen, but there is a great economic incentive for work to be done in this field.

Drying of coating is an area in which there should be additional work. Drying is not a major problem on board machines as they are run today. However, there is no uniform system of drying, nor does there seem to be an authoritative source on this subject. More must be learned on drying in order that on machine coaters might be used with higher efficiency with less machine space.

There is great opportunity for the suppliers of elastomeric materials to produce a superior binder for board coating. As more and more dark body stocks are coated, coating weight will increase. This requires a more flexible coating to prevent bender problems and dusting during cutting. Current elastomeric materials give good flexibility but they tend to reduce opacity, and some of these compositions tend to give excessive yellowing if used in large amounts. There is a real challenge to the rubber and plastics industry to produce a binder which has all the good characteristics of casein or soybean protein, and in addition which is flexible. For widespread use, this material should sell in the range of soybean protein.

One of the cost factors of coated board is that coatings add to the weight per square yard of board, and therefore, increase the cost for a given caliper. Much of the weight of the coating comes from pigment, and titanium dioxide is a very expensive pigment to use in a paper product which sells for a few cents per pound. One solution to this is to eliminate pigment from the formulation and to use dispersed air to give hiding power. There are specialty coated papers on the market today involving this principle and its use in coated board may not be too distant in the future.

Received May 27, 1959. Presented at the Tenth Coating Conference of the Technical Association of the Pulp and Paper Industry, Boston, Mass., May 25–27, 1959.

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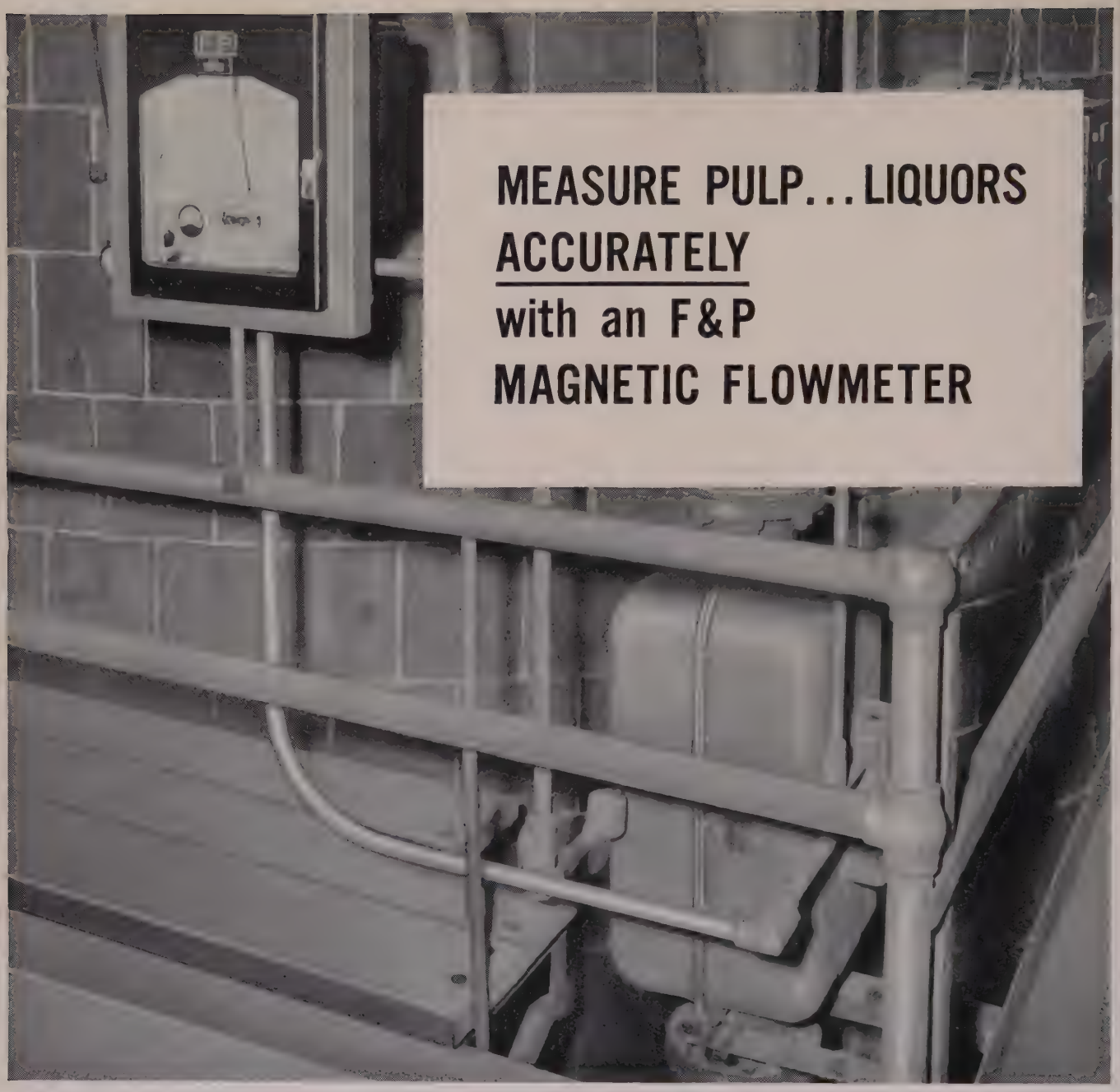

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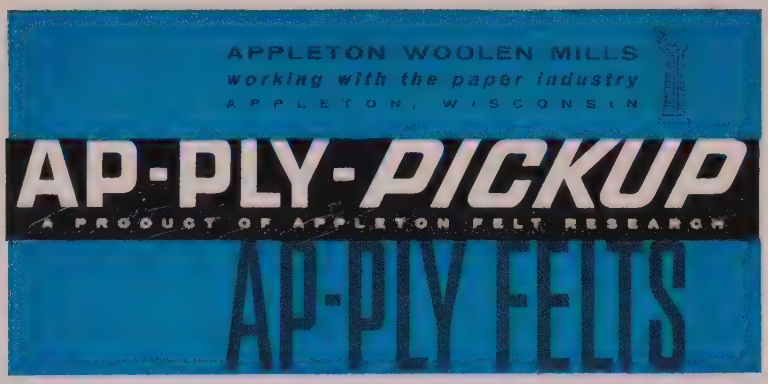

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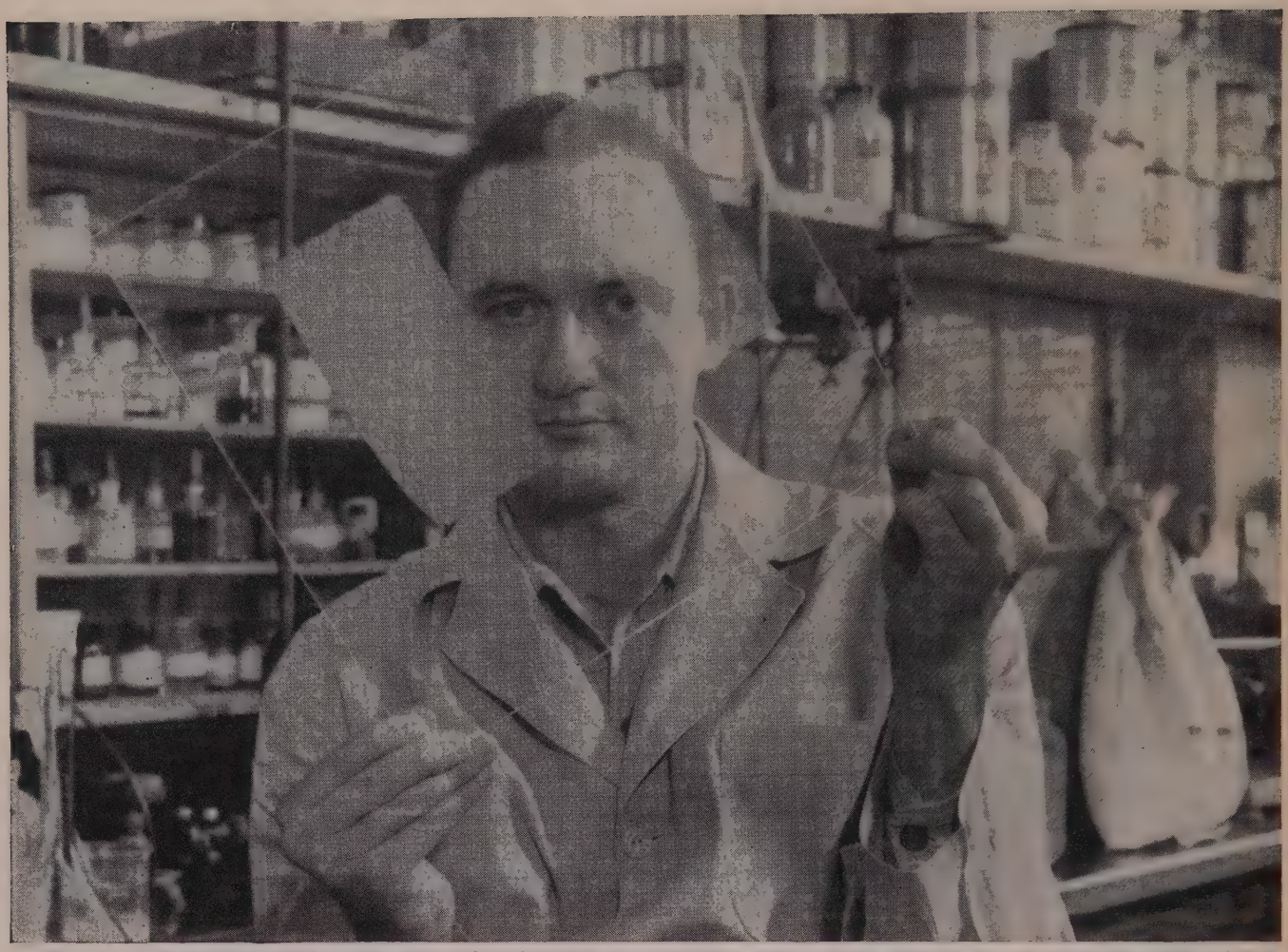

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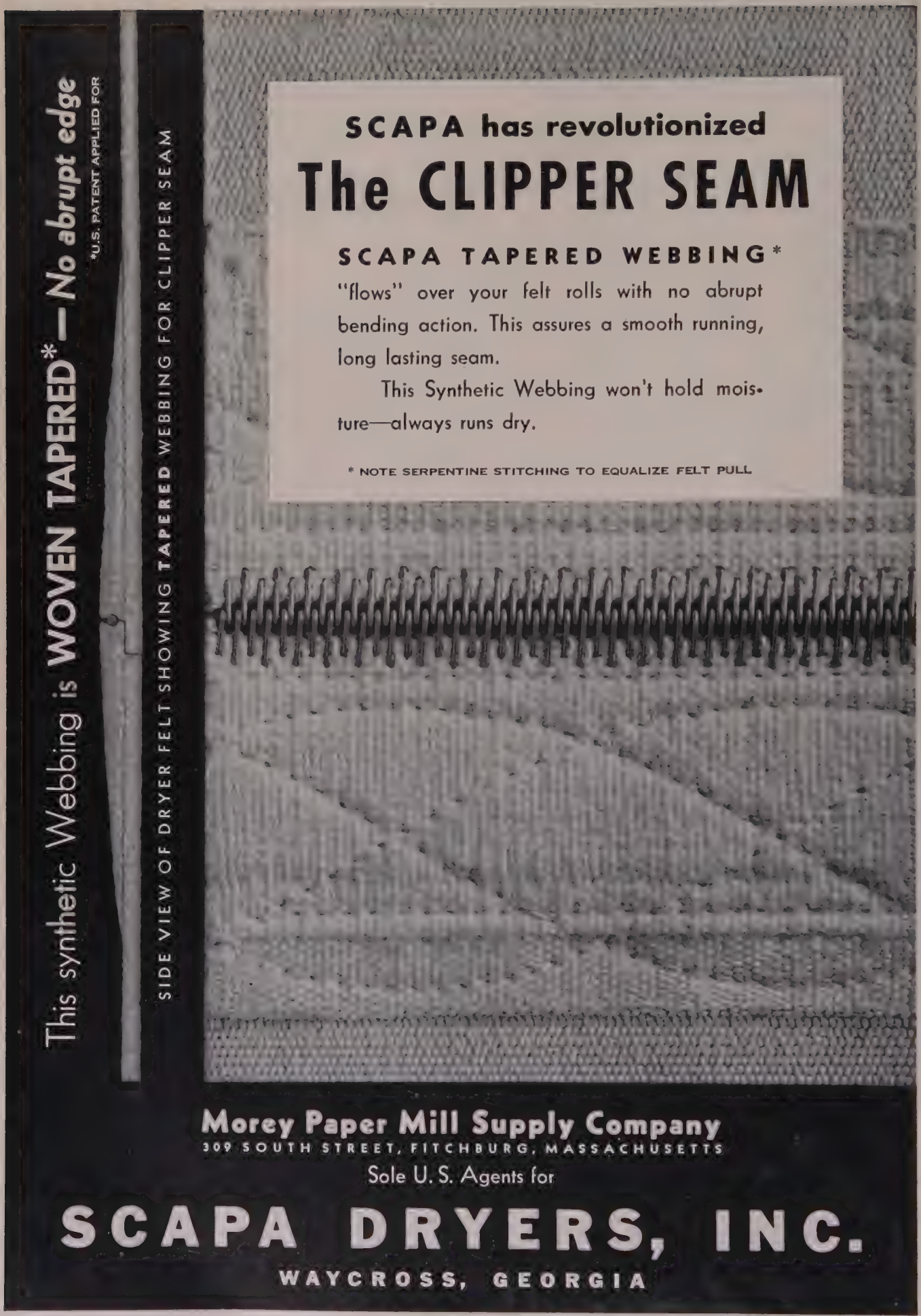
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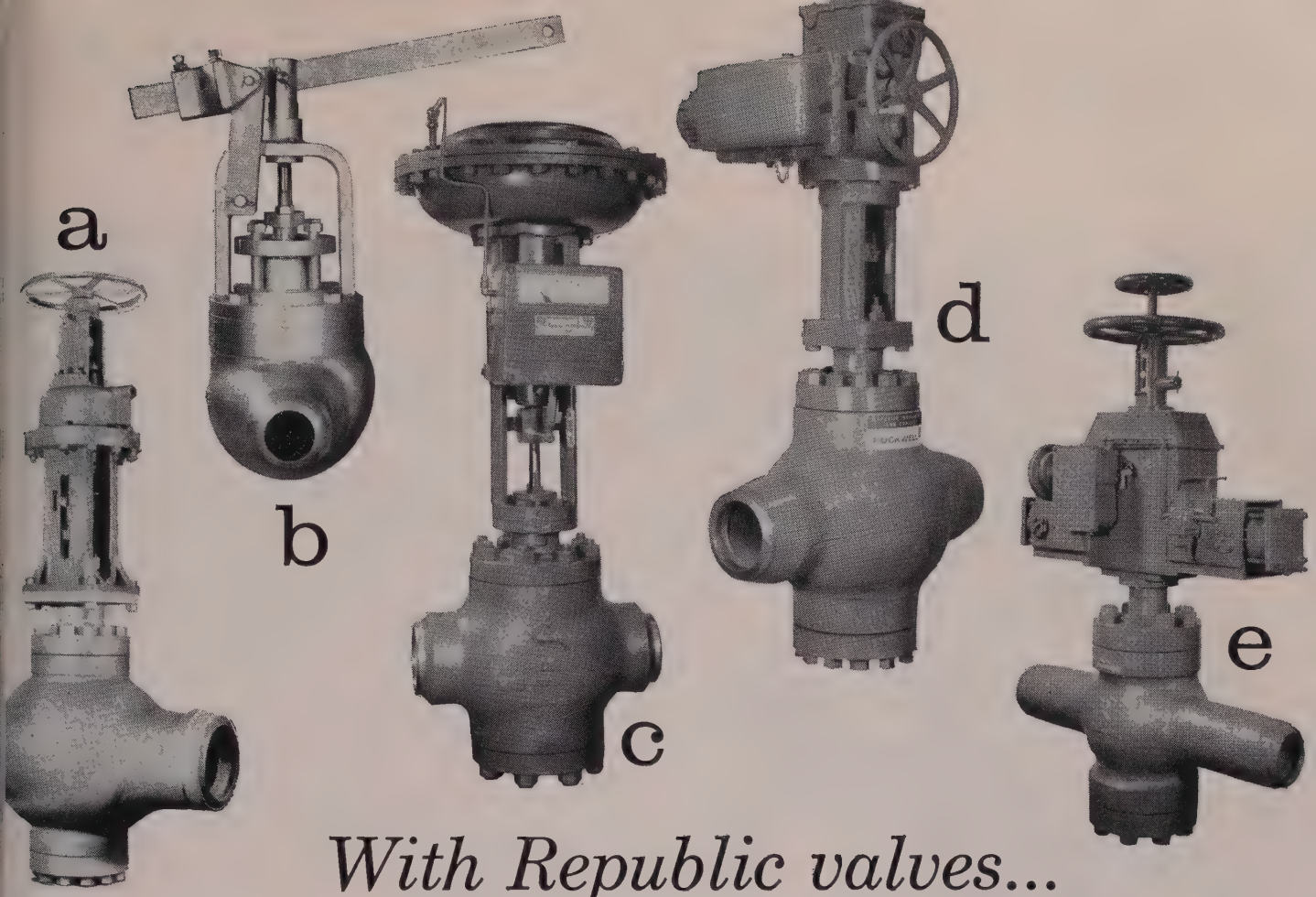

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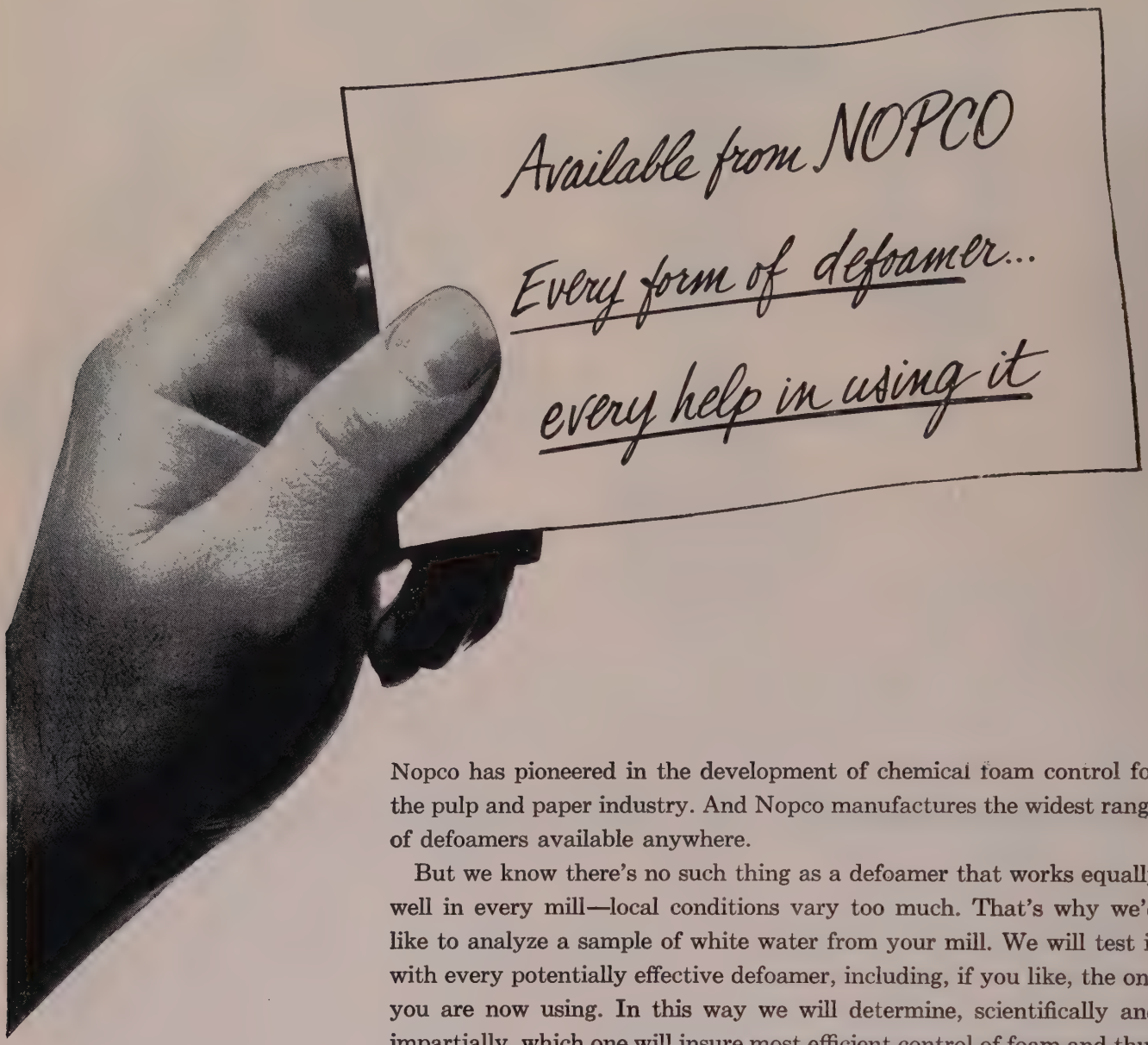

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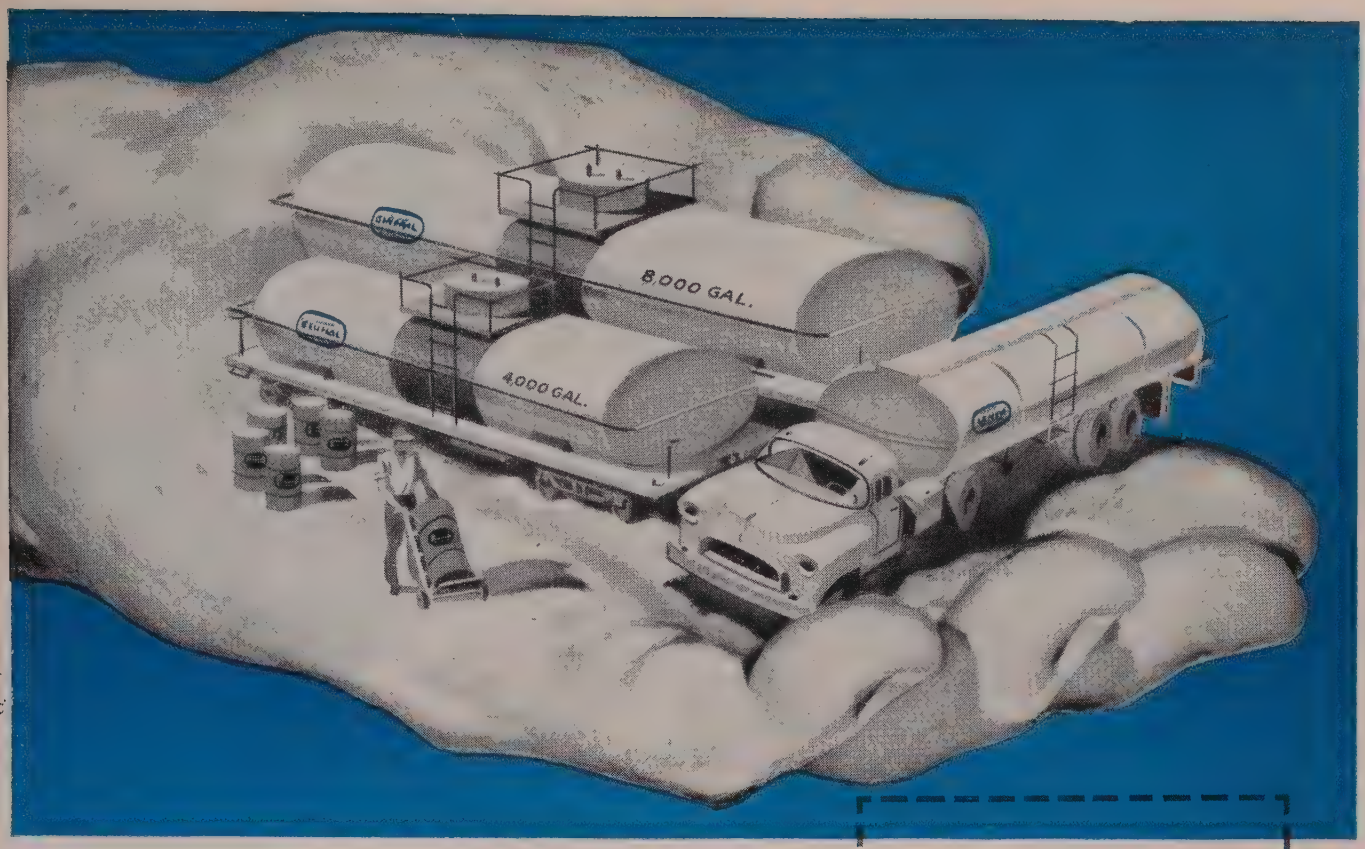

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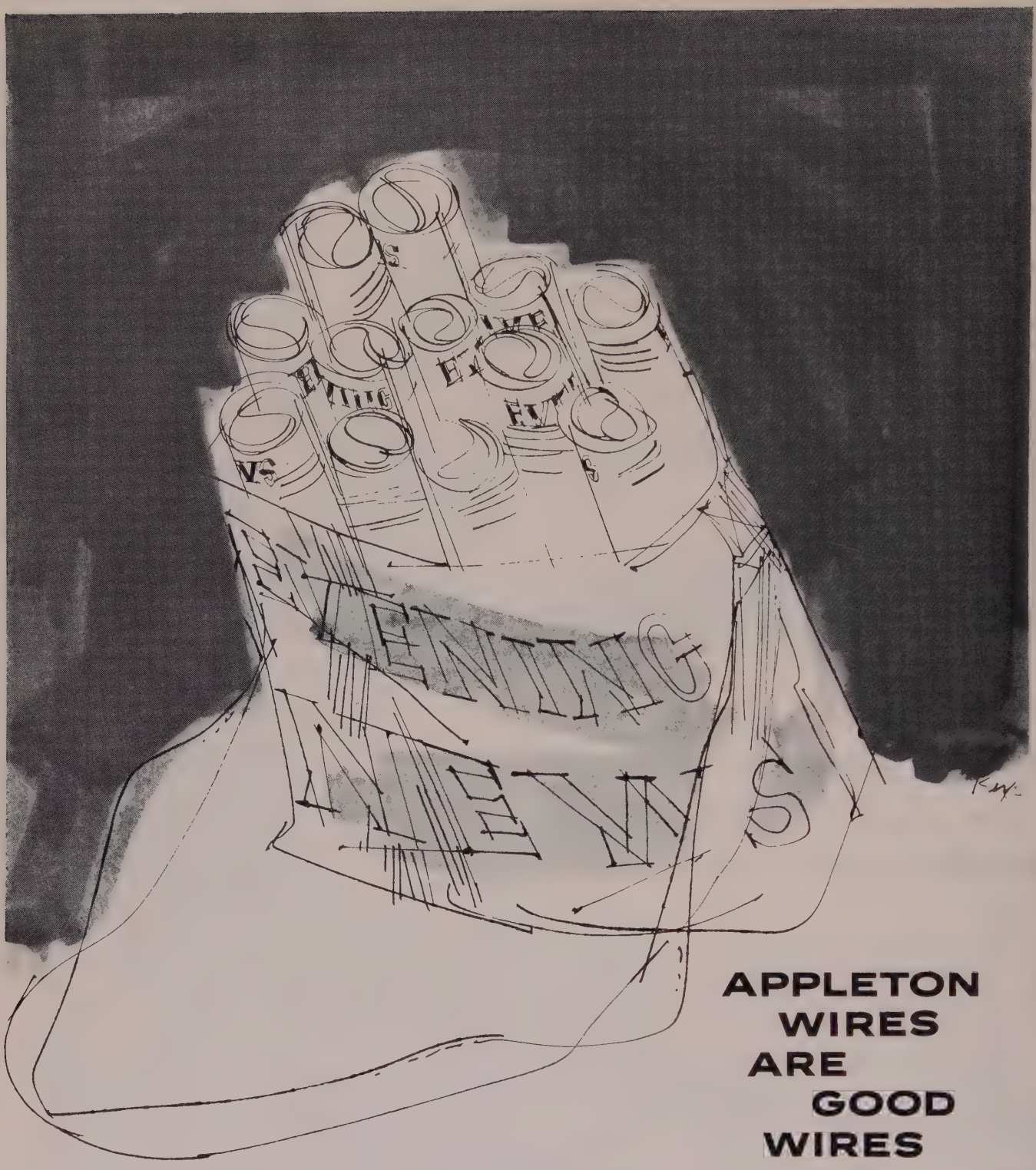

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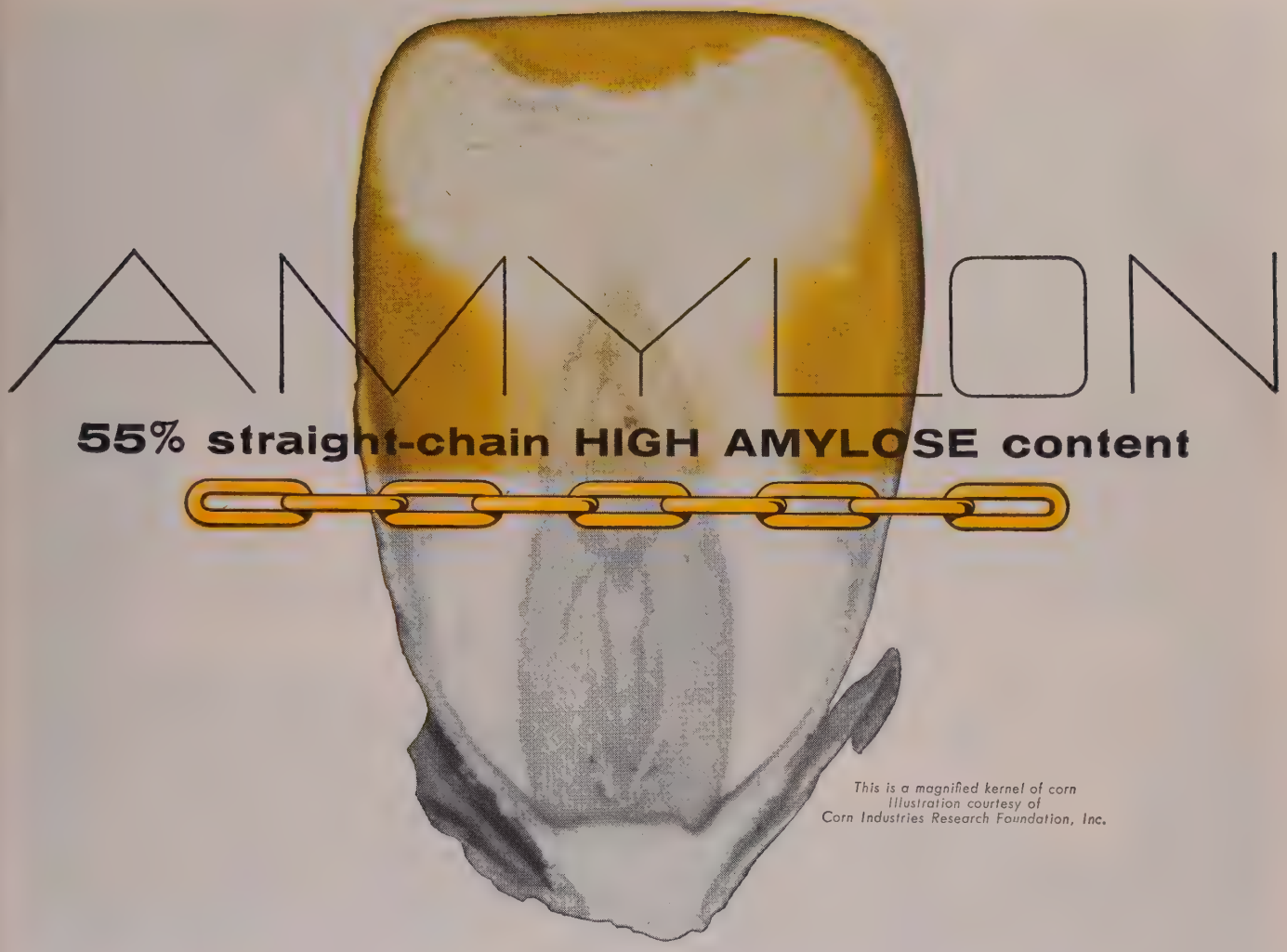

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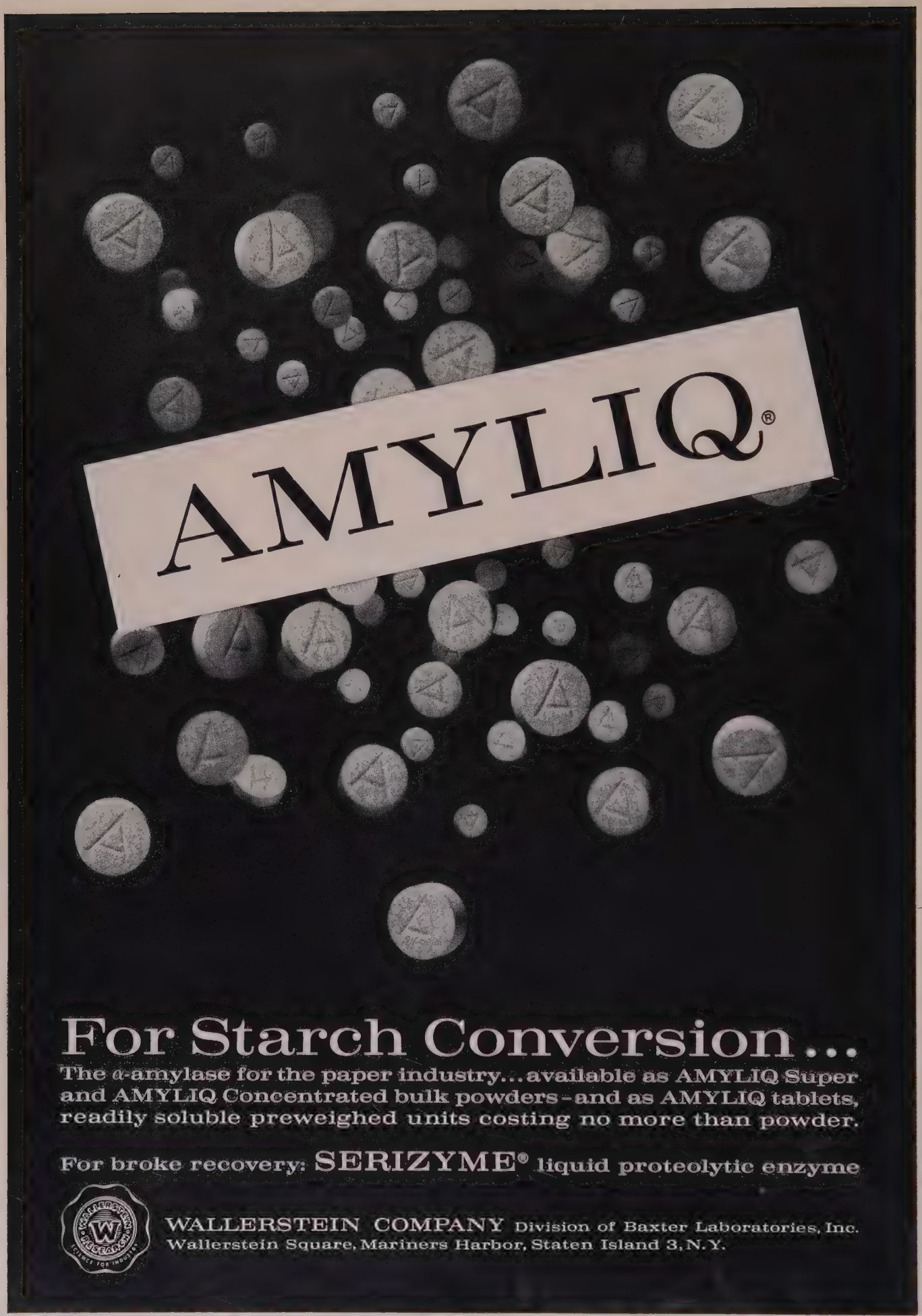

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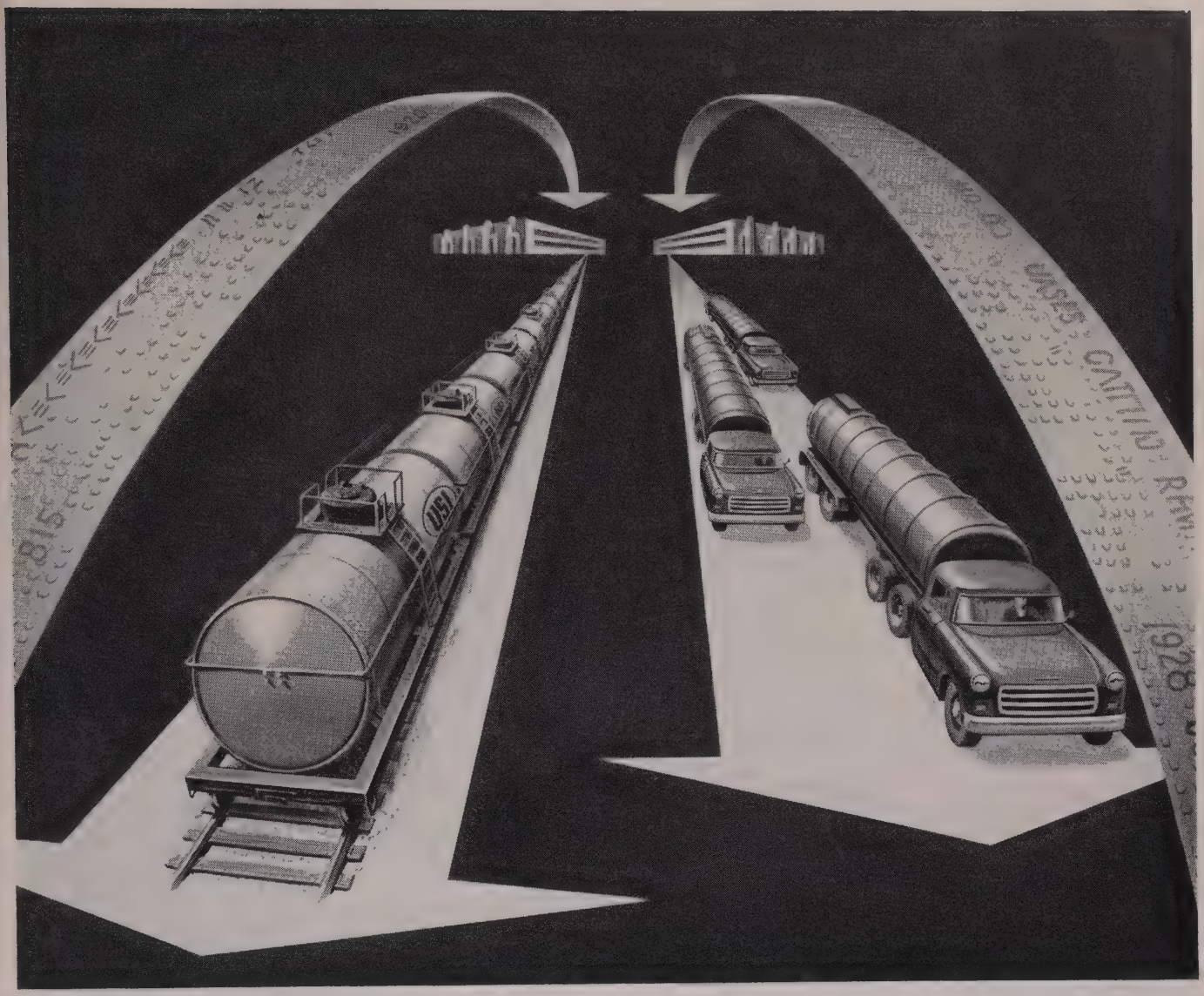

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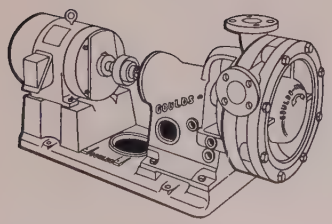

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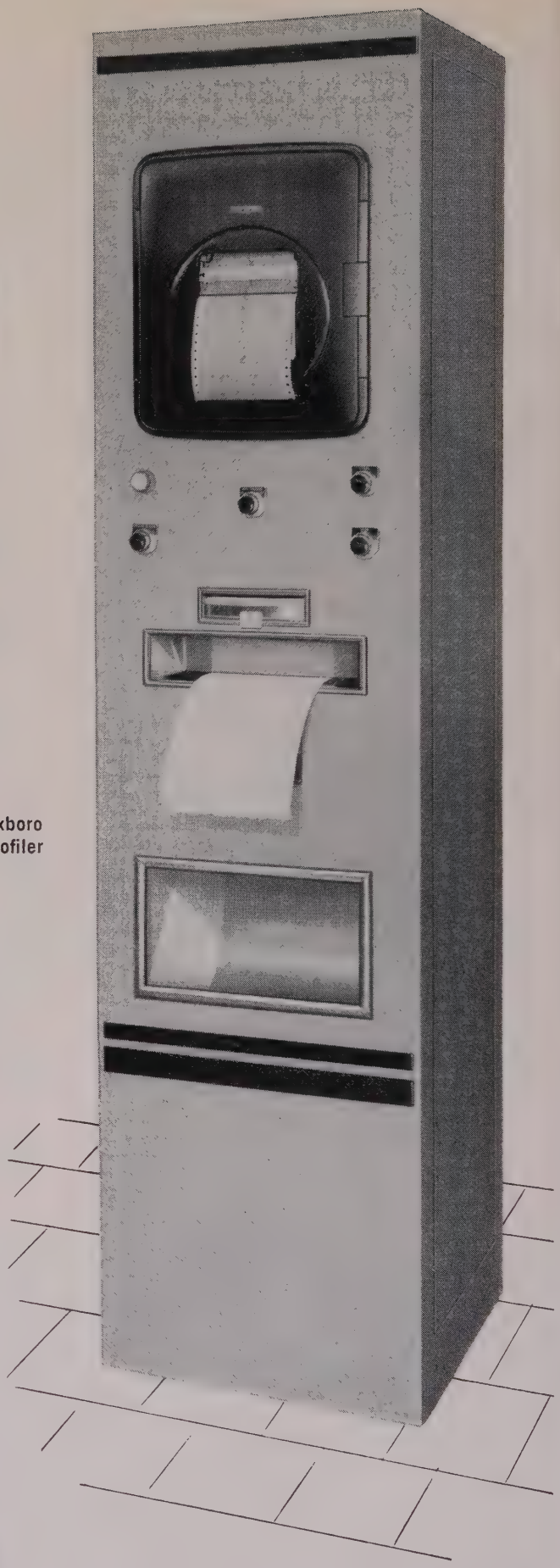

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INSTRUMENTATION FOR THE PULP AND PAPER INDUSTRY

What Are the Wood Properties Required by the Paper Industry in the Trees of the Future?

H. E. DADSWELL, A. J. WATSON, and J. W. P. NICHOLLS

The separate effects of cell length, cell wall thickness, pentosan content, and lignin content have been determined in each case keeping all other factors constant. In this way the marked increase in tearing strength with increase in cell length has been established; the effect of cell wall thickness on pulp strength properties other than tear have been further emphasized with pulps from three separate species of *Eucalyptus* in which the average cell lengths were identical; a wide range (7 to 15%) in pentosan content of hardwood pulps can be tolerated without loss in pulp strength although with reduction in yield at the lower values; for best pulp strength complete elimination of lignin is highly desirable. In addition, it has been shown that in the softwoods and hardwoods under investigation the cellulose content (Cross and Bevan) increases through successive growth rings from the pith, reaching a more or less constant value after 6 to 8 yr., also that the cellulose content varies markedly (up to 10%) in comparable material from different trees of a species. It has been once more strongly emphasized that in tree breeding work for the paper industry cell length, which is correlated with cellulose content, and basic density, which is a good index of cell wall thickness, are characteristics that must be taken into account. A method of determining wood properties in those trees selected by foresters as possible parents has been briefly described, and examples given of the way each tree can be assessed in relation to the average and extremes for the species.

ATTENTION has been directed previously (*1*) to the lack of specific knowledge concerning the relationships between pulp strength properties, wood structure, and chemical composition. The need for definite information was stressed because the forests of the future are being planned and planted *now* in many parts of the world. With knowledge available on just what is wanted in the raw material it should be possible to grow such material with the structure and chemical composition that is required. Unfortunately, because of the general lack of concern by the industry as a whole in its raw material, there is insufficient knowledge about the influence of wood characteristics on pulp strength properties. In the paper referred to (*1*) the position at the time was summarized and the wood qualities considered most desirable from the papermakers angle listed. The main conclusions were:

1. From the evidence it appeared that cell structure, in particular cell length and wall thickness, had an important bearing on pulp strength properties. Although an increase in cell length in any one case was associated with an increase in several strength properties, in particular tearing strength, there was some difficulty in isolating the length factor from other factors in the experiments carried out—namely pulping of wood from successive growth rings from the pith in a young tree of *Pinus radiata* (*2*), and one of *Pinus taeda* (*3*). The suggestion was made, therefore, that further information on the effect of cell length was desirable. Most of the evidence was obtained from work on softwoods; insufficient data were available on the cell length factor in the case of hardwoods.

On the other hand, some definite information is available on the influence of cell wall thickness through the work of Runkel (*4*) who pointed out that thin-walled fibers in which $2w/1 < 1$ (w = wall thickness, l = lumen diameter) are the best because they collapse, become ribbonlike, and provide a large surface area for bonding. Thick-walled fibers do not collapse, are stiff, and retain their rounded shape during sheet formation; although they do not bond readily and are resistant to beating they increase the tearing strength. Sheets made from thin-walled fibers of equivalent length are much weaker in tearing strength, but are superior in other properties.

2. Basic density (based on the ratio of oven-dry weight to volume green or soaked) provides an excellent clue to the suitability of a timber with respect to the properties of the pulp to be derived from it. It gives an indication of cell wall thickness, particularly in hardwoods where an increase in wall thickness is reflected in a higher basic density. In those softwoods where there are well-defined growth rings an increase in density is closely correlated with an increase in percentage late wood, which of course means an increase in the number of thicker-walled fibers. For certain grades of paper, wood with a low basic density is most desirable; on the other hand a high density means a high yield and a greater tearing strength.

3. Reaction wood: tension wood in hardwoods and compression wood in softwoods, is undesirable and should be eliminated wherever possible. Its effects on pulping and pulp strength have been discussed elsewhere (*5*).

4. Insufficient is known about the influence of chemical composition on pulp strength properties, in spite of some 40 years research on the chemical composition of wood. In general it would appear that the pentosan content of the pulp has an important bearing on the development of strength (*6*). However, the exact amount desirable in any one pulp for best results is not known; freedom from hemicelluloses in general is a desirable characteristic for dissolving pulp. The presence of lignin hinders pulp strength development, and therefore for best results it has to be removed during the pulping process or subsequent treatments. The ideal wood is one with a low lignin content and little or no extractives because both of these components consume chemicals during their removal and cause lower yields of pulp.

H. E. DADSWELL, Assistant Chief, A. J. Watson, Senior Research Officer, J. W. P. NICHOLLS, Experimental Officer, Division of Forest Products, Commonwealth Scientific and Industrial Research Organization, Melbourne, Australia.

From the analysis of the available evidence the following wood qualities were selected as most desirable in wood to be converted into paper:

1. Higher than average fiber length for the species;
2. Cell wall thickness such that $2w/l < 1$;
3. Basic density lower than average for the species (this is correlated with (2));
4. Higher than average cellulose content;
5. Low extractive content;
6. Suitable pentosan content in the pulp (which has to be determined for each species);
7. A growth rate as rapid as the economics of plantation management will allow.

It is not only necessary to have a list of desirable features, it is equally necessary to know whether these features can be transmitted from parent to progeny in tree breeding. There is every reason to believe that cell length and basic density are genetic features (*7*, *8*). Accepting that this is so we have developed a method for the assessment of these features in living trees, and from the results obtained can advise as to which of selected trees are the most satisfactory for tree breeding from the point of view of wood characteristics (*9*). This method is described briefly below.

Because of uncertainties revealed in the investigation of wood properties in relation to pulp strength, we have in this laboratory over the past 12 months deliberately set out to ascertain what influence selected features of wood structure and chemical composition might have. In this work material from young and mature trees of selected softwoods and hardwoods has been used. It could not, of course, be expected that answers to all the queries would be forthcoming in such a short period; however, some most interesting preliminary results have been obtained and although our work is by no means complete it is possible to give here a resume of what has been found to date.

THE INFLUENCE OF FIBER STRUCTURE

Cell Length

As is well known (*10*) there is a marked increase in cell length through successive growth rings from the pith at any one level in the tree. After a number of years, varying from species to species, a more or less constant value is reached. Usually the period is from 12 to 15 yr. in plantation-grown softwoods in Australia and somewhat longer in naturally regenerated eucalypts. The rate of growth of both the plantation-grown softwoods and the eucalypts may be such that the early growth rings (1 to 6+) are quite wide—often $^1/_2$ in. or more. This means that there is sufficient wood in individual growth rings for pulping under laboratory conditions. This has been done for *Pinus radiata* (*2*) and *P. taeda* (*3*), and in each case a series of pulps, cooked to a comparable degree, obtained. From these pulps it was possible to get some idea of changes in pulp strength properties with changing cell length. The most important result observed was this correlation between cell length and tearing strength over the early growth rings where the changes in length were greatest.

It was evident, however, that other factors which accompany the change from juvenile to adult wood were clouding the picture. Therefore, a further experiment was devised in which cell length only was varied while all other factors were kept as constant as

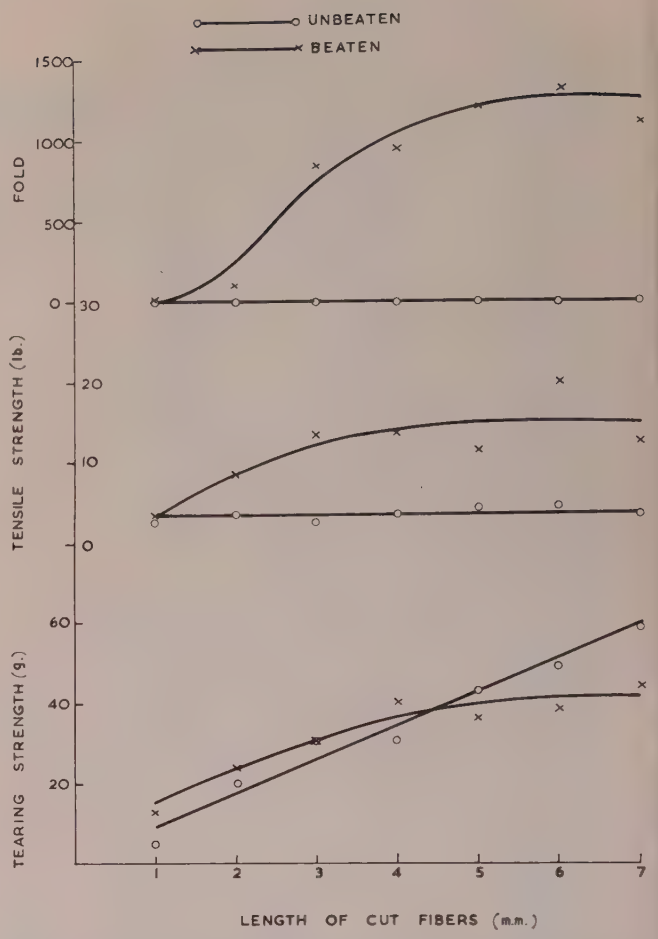

Fig. 1. Showing the variation in pulp strength properties—unbeaten and beaten—in relation to length of fiber. The "fibers" used were pieces from Araucaria klinkii fibers cut to predetermined lengths

it was possible to do. The idea was to select a species with abnormally high average cell length in mature wood and to cut the fibers into selected lengths before determining pulp strength properties. This was done using the wood from the outer growth zones of a mature tree of *Araucaria klinkii* in which the average cell length was found to be 8 mm. Chips approximately $1^1/_2$ in. wide by $1^1/_2$ in. long (tangential face) and $^1/_8$ in. thick (radial direction) from the selected growth zones were mixed and then delignified by treatment with sodium chlorite. These delignified chips would not separate into individual fibers until given a treatment with dilute sodium hydroxide. It was therefore possible to cut them into any desired length before maceration using a hand guillotine. In this way numerous pieces randomly taken were obtained in lengths of 1, 2, 3, 4, 5, 6, and 7 mm. Each batch of pieces of the same length was then macerated giving a number (seven) of "pulps" in each of which the upper limit of cell length was definitely known. All material was comparable in every other characteristic with the exception of "cell" length. Microscopic examination of the pulps revealed that, in each case, a very uniform length distribution had been obtained.

Each batch of "pulp" suspensions was used to make handsheets using unbeaten material and material beaten in a small high-speed laboratory stirrer for 30 min. The handsheets from the unbeaten pulps needed special handling techniques for couching and separation

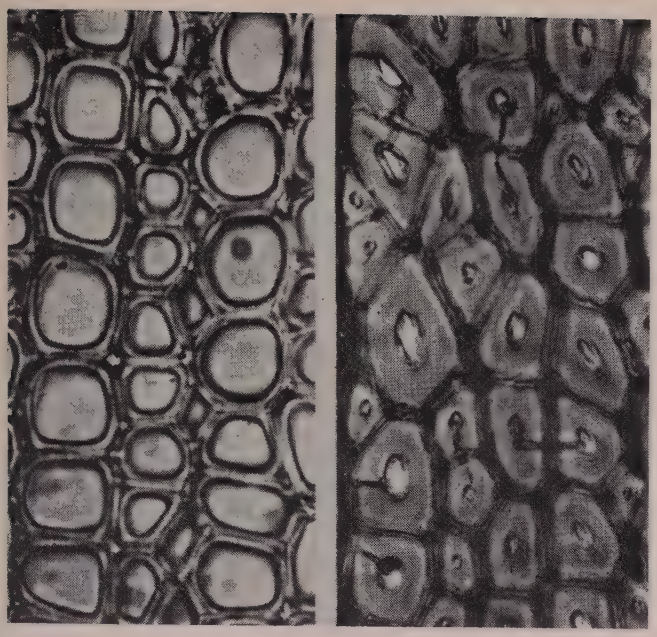

Fig. 2. Cross sections of **Eucalyptus regnans** *(left) and* **Eucalyptus diversicolor** *(right) showing the variation in cell wall thickness in these two species.* × *640*

from the blotters; nevertheless, sheets suitable for some pulp strength determinations were obtained. The results for tearing strength, tensile strength, and folding endurance for the pulps of different average "cell" lengths are shown in Fig. 1.

The low tensile strength and folding endurance values for the unbeaten "pulps" indicate that their bonding is very poor, and that it is not affected by the "cell" length. However, tearing strength showed a direct linear relationship with "cell" length; a very definite increase in strength being associated with increase in length. As in the present case fiber bonding appears to play only a minor role, it is clear that "cell" length is the dominant and perhaps the only factor governing tearing strength.

The same general conclusions apply with the beaten pulps, but here the greatly improved fiber bonding due to beating complicates the interpretation of the data, as it is difficult to separate the influence of bonding from that of "cell" length. However, it appears that increase in cell length causes improvement in both folding endurance and tensile strength providing a certain degree of bonding is obtained. These results are certainly of considerable value but they must be followed up by other investigations. Nevertheless one can, we believe, be fairly certain *now* from the experimental evidence that within a species any increase in cell length is most desirable from the papermaking point of view. Therefore, the tree breeder should without hesitation strive to develop in the selected species strains with higher than average cell length.

Wall Thickness

The work of Runkel (*4*) on the importance of cell wall thickness in papermaking is well known. In our recent investigations we have been able to demonstrate the effect quite definitely using composite wood samples from three different species of the genus *Eucalyptus*, namely, *E. regnans* F. Muell., *E. marginata* Sm. and *E. diversicolor* F. Muell. The woods of these species vary markedly in density, the averages being:

E. regnans (mountain ash)—basic density 30.0 lb. per cu. ft.
E. marginata (jarrah)—basic density 40.5 lb. per cu. ft.
E. diversicolor (karri)—basic density 44.0 lb. per cu. ft.

In these species the area occupied by vessels and parenchyma is much the same so the variation can be attributed to differences in cell wall thickness. This is confirmed by microscopic examination of cross sections, examples of which are shown in Fig. 2 (a and b).

Pulps from the composite wood samples of each of these species were prepared by the sulphate process using similar conditions and cooking to a comparable lignin content. Strength properties of handsheets prepared from unbeaten and beaten pulps were then determined; results are given in Table I. As each pulp had similar lignin contents and as the average fiber lengths were identical (1.31 mm. in each case) it was possible to consider from these results the extent of the influence of wall thickness. Admittedly, the pentosan content of the pulp from *E. marginata* was somewhat lower than that of the other two pulps (13% versus 17.5%) but as will be seen later this may not be as significant as previously thought.

There was little difference between the strength properties of *E. marginata* and *E. diversicolor* pulps, although the tearing strength figures of the former were at all stages of beating slightly superior. Indeed, all three pulps gave excellent tearing strengths for hardwoods, a fact which can to some degree be attributed to the cell lengths. The pulp from *E. regnans* was su-

Table I. Evaluation Data on Sulphate Pulps Prepared from Composite Samples of *E. Regnans*, *E. Marginata* and *E. Diversicolor*

(All pulps prepared using the same cooking conditions and beaten in the Lampen mill)

	Beating, rev.	*Bulk*	*Burst factor*	*Breaking length, km.*	*Tear factor*	*Air resistance, sec.*	*Fold, K.M.*	*Freeness, C.s.f.*	*Drainage time sec.*
E. regnans	0	1.79	36	6.6	109	5.2	30	464	5.4
	1,125	1.62	49	8.6	135	5.4	81	478	5.8
	4,500	1.50	68	10.0	133	11.0	401	458	6.1
	9,000	1.42	78	10.8	123	43.1	1734	363	7.0
	18,000	1.37	86	11.4	119	318.5	1932	211	10.1
E. marginata	0	2.04	19	4.4	70	1.2	8	453	5.4
	1,125	1.87	32	6.2	97	2.0	29	432	5.4
	4,500	1.71	46	7.9	117	6.9	145	291	6.2
	9,000	1.65	58	8.9	126	42.3	394	162	8.6
	18,000	1.57	66	9.6	129	185.9	1603	72	15.1
E. diversicolor	0	2.03	17	4.2	61	1.2	6	446	5.5
	1,125	1.81	28	5.7	87	1.6	8	457	5.6
	4,500	1.69	44	7.5	107	9.7	82	363	6.1
	9,000	1.58	55	8.4	112	61.5	368	206	8.8
	18,000	1.48	63	9.5	108	>20 min.	1494	89	15.7

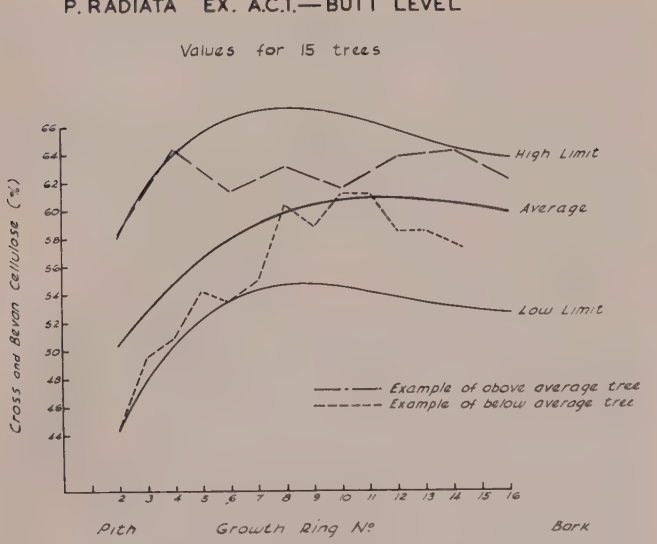

Fig. 3. Observed variations in cellulose content (Cross and Bevan cellulose) in successive growth rings from the pith of Pinus radiata. Average results for specimens from 15 trees from the Australian Capital Territory have been plotted as well as the high and low extremes. Results from two individual trees have been included for comparison with average and extremes

perior in tearing strength in the unbeaten and early stages of beating, and definitely superior in bursting strength and other properties at all stages. This superiority is attributed quite definitely to the better bonding capacity of the thinner-walled fibers.

The relationship between basic density and cell wall thickness in both hardwoods and softwoods (where there is a high correlation with percentage late wood) is quite definite within the one species. Between species there may be marked differences in vessel size and/or number, ray size and/or number, or amount of parenchyma, which would all influence density. However, from our results it would appear that in any selected species, particularly of hardwoods, breeding for low density would be most desirable for most strength properties. On the other hand, breeding for high density would increase yield and may assist tearing strength.

INFLUENCE OF CHEMICAL COMPOSITION

Pentosan Content

A series of pulps was prepared from a composite sample of a hardwood, *Eucalyptus regnans*, containing the same lignin contents but with pentosan contents varying from 19% to less than 1%. Handsheets were made from these pulps in the unbeaten stage and after beating in the Lampen mill for 18 min. This work will be reported in detail elsewhere (*11*) but the results revealed the low strength properties obtained using pulps with either a very high or a very low pentosan content. Bursting strength was at a maximum between 7 and 15% pentosan content; tearing strength at a maximum at approximately 8% for the unbeaten pulp and 5% for the beaten pulp. These results are of particular interest, but they need to be checked using other species of hardwoods and some softwoods. Nevertheless they do give a guide as to what is required in a hardwood pulp for best strength development.

Lignin Content

In a similar type of investigation using the same composite sample of *E. regnans*, the pentosan content of the pulp was kept constant while the lignin content was varied from 20 to 3%. It was observed that the presence of lignin was deleterious to all strength properties. For maximum strength development it is therefore desirable to have as little lignin as possible in the pulp.

Extractives

The presence of extraneous materials in the wood reduces yields and consumes chemicals in their removal. Therefore a raw material with little or no extractive content is most desirable. Some indication of the effect of extractives on yields of pulp was obtained in the work on the three eucalypt species referred to earlier. Percentages of materials soluble in alkali and yields are shown below:

	Alkali soluble, %	*Yield of pulp, %*
E. marginata	23.7	45.4
E. regnans	18.1	52.0
E. diversicolor	16.1	56.0

Cellulose

It is axiomatic that materials with high cellulose content are most desirable for pulping. By "cellulose" we mean the resistant polysaccharide portion which has been designated as Cross and Bevan cellulose. The amount present in wood varies considerably between species, within a species, and within a tree. Large tree to tree variations in water-resistant carbohydrates and alpha-cellulose were observed in loblolly pine by Zobel and McElwee (*12*) who also referred to differences in cellulose content between juvenile and mature wood and the fact that trees with low yields in juvenile wood also had low yields in mature wood and the converse.

In earlier work in this laboratory (*13*) and recently it has been shown that the cellulose content (Cross and Bevan) varies through successive growth rings from the pith in a manner similar to that observed for cell length. This has been confirmed in the examination of samples from 15 trees of *Pinus radiata* and average results for all trees have been plotted in Fig. 3 against growth ring number from the pith. At the same time, in the same figure the limits of variation of the extreme values, high and low, have been shown. The important

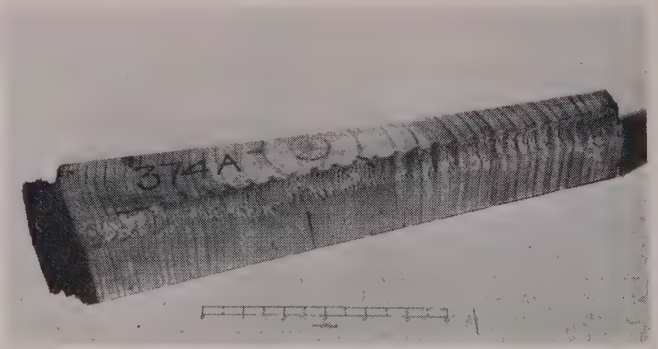

Fig. 4. The type of specimen taken from a selected live tree for use in the determination of various wood characteristics

fact that emerges is that some trees of a species have a higher than average cellulose content which should make them desirable as parents in tree breeding work. Where this is the case the high cellulose content is maintained through successive growth rings, thus confirming the observations of Zobel and McElwee on juvenile and mature woods.

In the 15 trees examined there was a highly significant correlation (1% level) between cellulose content and fiber length. This is due no doubt to the increase in both with age, but it is a fact that is important because fiber length can possibly be controlled genetically, and it would be most advantageous if high average fiber length also means high cellulose content.

From the pulping investigations carried out on specimens taken from successive growth zones from the pith in *E. diversicolor* (in which species detection of growth rings is most difficult) a similar problem of change in cellulose content within a tree and between trees has been observed. The actual difference within a tree was not as marked as in the *P. radiata*. This may well be related to the smaller percentage increase in cell length (30 to 40% in the case of a eucalypt timber as against 200 to 300% in the case of a softwood).

For this work a semimicro method of cellulose determination has been developed. Details of it will be published elsewhere. It has been necessary to have such a method because of dealing with quite small quantities taken from living trees. For work of this kind, therefore, some of the more usual wood analytical methods will have to be modified. This is a challenge to the wood chemists.

ASSESSMENT OF WOOD CHARACTERISTICS IN LIVING TREES

An important point to consider is just how the required structural features and chemical composition can be determined and assessed in those trees selected by the forester as most desirable parents on external

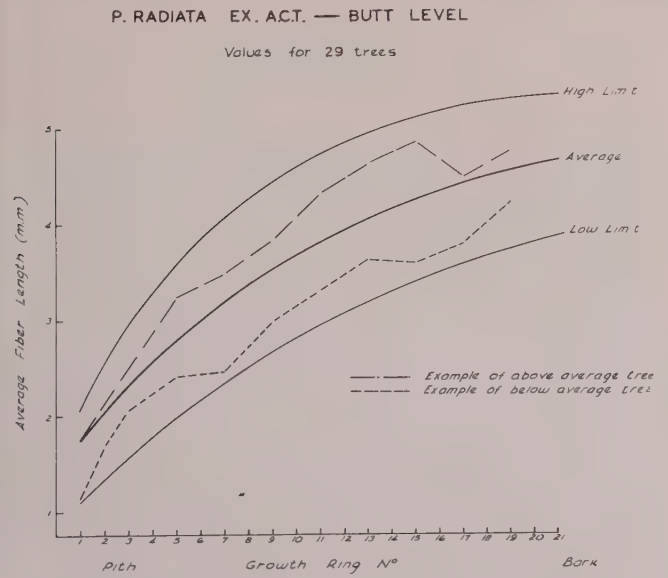

Fig. 5. Variation in fiber length through successive growth rings from the pith in* Pinus radiata. *Average and extreme values have been plotted using results obtained from the examination of specimens from 29 trees of the species selected in the Australian Capital Territory. Examples from below average and above average trees are shown in relation to the average and extremes

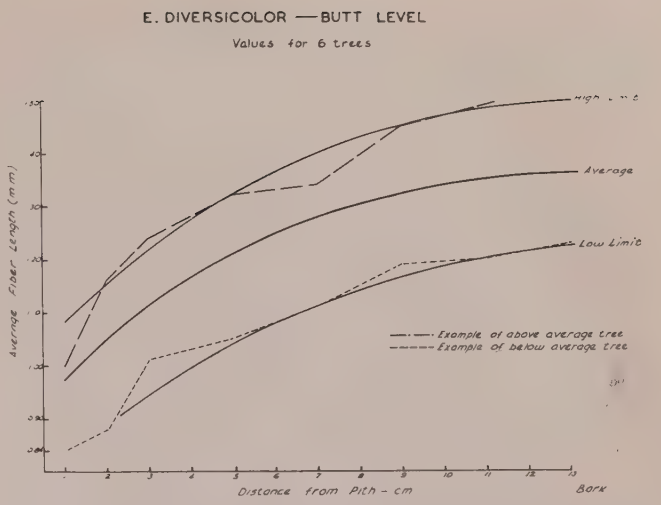

***Fig. 6. Similar to Fig. 5, but showing fiber length variations with distance from pith in the case of* Eucalyptus diversicolor**

features. Because our future supplies of raw material for the industry will, in most parts of the world, be derived from young, fast grown softwoods and hardwoods, we consider it necessary to sample the selected trees for the examination of wood characteristics over the whole of the growth range, i.e., through successive growth zones from pith to bark. Therefore, in this laboratory a method of sampling and examination of the wood has been developed with the object of getting the maximum amount of information from the smallest possible specimen. Full details of this method are published elsewhere (*14*) but they have been summarized briefly here.

Wedge-shaped specimens, approximately $1^1/_2$ in. wide and 2 in. long (tangential face), are cut out along a diameter of the standing tree from bark to bark, according to the procedure originally outlined by Echols and Mergen (*14*), but recently modified by Brown and Fielding (*15*). The specimens as received in the laboratory are in the form shown in Fig. 4. Care must be taken to ensure that the specimens are cut at a constant height above ground in all trees of a species sampled. In our work the selected height was 3 ft. 6 in. It is also essential in comparative work of this type to use the same radius for each tree because of the possibility of some variation in cell length around the one growth ring. Reference has been made to this possibility in the work of Liese and Dadswell (*16*) who have shown that in trees grown in the southern hemisphere the lowest average cell length in a selected growth ring is apt to be found on the north or northwest side of the tree, whereas in the northern hemisphere it is found on the south side of the tree. Hence in our work the wood along the north or northwest radius is used.

In the laboratory the specimens so obtained are used for the determination of cell length, micellar angle, basic density, percentage late wood, longitudinal and tangential shrinkage, cellulose content, maximum crushing strength, etc., using wood from successive growth rings or zones from the pith. Results so obtained are average for the trees sampled from a particular area, and the average values plotted against growth ring number. For assessment purposes the high and low values are also plotted on the same graph (see Figs. 5 and 6). Results for individual trees can then be

plotted to the same scale and compared with the average and extreme. Examples of what has been done in the case of fiber length for trees of *P. radiata* and *E. diversicolor* are given in Figs. 5 and 6. It will be apparent that there is a wide difference between the extremes and that it is possible by the method outlined to select those trees with higher than average cell length (or any other selected characteristic) for the species. It has been applied too in the case of cellulose content (see Fig. 3).

This method of attack is relatively simple, although the collection of data takes time and patience. However, from the results it is possible to give a very good lead to the tree breeder regarding desirable wood characteristics. The success of the whole procedure will depend, of course, on the degree to which selected characteristics may be transmitted from parent to progeny. In this regard present evidence indicates that both cell length and specific gravity can be genetically controlled. From the work carried out in this laboratory, these emerge as the two most important wood features, both for timber utilization and from the point of view of the papermaker. It is therefore strongly recommended that they be given a high priority in tree breeding work.

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Tensile Strength Changes Due to Hypochlorite Bleaching

L. F. McDONNELL and M. N. MAY

Jappe (1) reported that the application of hypochlorite bleach, under certain conditions, to NSSC aspen pulp affected the strength properties through relatively few chemical changes in the pulp. This knowledge was exploited in a study of some of the factors which influence the tensile strength of paper. The only significant chemical changes detected were delignification and a decrease in the D.P. of the pulp. Greater bonding strength of the bleached pulp was attributed to an increase in surface area resulting from deshiving, fiber swelling, and fibrillation. It also appeared that bleaching provided a surface better capable of bonding. Fiber strength was reduced by hypochlorite, and a nonlinear correlation is shown between fiber strength and pulp D.P., although there is some reservation about unqualifiedly accepting the relationship. The conditions and range of the study were chosen to show that tensile strength is a function of a balance between fiber strength and interfiber bonding.

Recently, Jappe (*1*) studied the effects of sodium hypochlorite as a third stage in the bleaching of neutral sulphite semichemical (NSSC) aspen pulp. Although the pulp constituted a complex system, Jappe found that the chemical changes attributable to hypochlorite treatment were relatively few. The primary effect was delignification with a small reduction in the average D.P. of the pulp. There was no significant change in the amount or D.P. of the hemicelluloses. Accompanying these chemical changes was a decided increase in the tensile and bursting strength of papers produced, without additional beating, from the bleached pulp. This system appeared to offer a relatively simple system for studying more completely some of the factors which influence the strength of paper.

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FACTORS INFLUENCING PAPER TENSILE STRENGTH

In the papermaking process, most of the water is removed by filtration and pressing, leaving an agglomeration of the wet fibers. As the remaining liquid is removed by evaporation, surface tension forces draw the fibers and fiber elements together. In the final stages of water removal, hydrogen bridges are established between the alcoholic hydroxyl groups of neighboring fibers and fiber elements, thus uniting the fibers.

Many investigators have recognized that fiber-to-fiber bonding is an important factor in the strength of paper (*1–4*). Lignin is of considerable significance in this respect because of its adverse effect on bonding (*1, 5, 6*). In part, this is presumably because it is a partner in fewer and weaker bonds. Lignin also may contribute to impaired fiber flexibility or conformability because the outer wall of the fiber, which is interpenetrated with lignin, may act as a sheath on the fiber retarding the imbibition of water, and thus restricting swelling. Many past studies (*7–9*) have been designed to show the effect of lignin on paper strength. It has been difficult to draw unqualified conclusions from most of these because of other changes as the lignin content was varied.

The role of hemicelluloses in paper has been debated at length. Cottrall (*10*) and Greenane (*11*) have observed that their presence is coincident with strong papers. Jayme and Roseneld (*12*) postulated that the more highly swelling hemicelluloses in sulphite pulp are concentrated in the outer layers of the fiber. Hemicelluloses may act by one or both of two mechanisms. The proponents of the "mucilage" theory assume that the hemicelluloses swell by imbibing water to form a colloidal mucilage which tends to glue the fibers together, presumably through hydrogen bridges. On the other hand, hemicelluloses are conceded to imbibe water to a greater extent than cellulose, so that by their presence within the structure, the fiber may take up water and swell. Such swelling could make the fibers more flexible and conformable. At the same time, and perhaps with the added impetus of some mechanical action, some of the fibrils and cellulose chains, particularly those in the noncrystalline regions, are freed from close association with the fiber. Then, when water is removed from the paper structure, the flexible fiber elements may more readily conform to the influence of surface tension forces so that hydrogen bridges may form between the alcoholic hydroxyl groups of the cellulose chains.

The degree of swelling and flexibility of the fibers probably affects the conformability and therefore influences the development of bonding. Giertz (*13*) stated that thin-walled fibers produce a sheet with higher tensile strength than thick-walled fibers and suggested that this was because of greater flexibility and collapsibility, resulting in larger contact surfaces. McKenzie and Higgins (*14*) caused pulp to swell in pyridine and noted a 50% increase in papers made from the swollen stock.

The tensile strength of a sheet of paper increases with increasing fiber bonding until the strength of the fibers themselves becomes the limiting factor. The actual strength to anticipate in fibers is difficult to estimate. Mark (*15*) calculated the limit of tensile strength to be 51,000 m., based on the assumption of completely crystalline cellulose. This value does not account for the reduced density and alignment of cellulose chains, nor the fact that stress concentrations and imperfections may be responsible for lower values.

Relationships between fiber strength and D.P. of cellulose have been demonstrated for textile fibers (*16*), but the same general relationship has not been shown for paper pulp fibers (*13*). Cottrall (*10*) has stated that the data available to him indicated that so long as pulp viscosity is above a certain minimum, it has no relationship to the papermaking strength (tensile) of the pulp. The inference, from published speculation, is that if the D.P. of cellulose in the fibers is greater than about 1200, variations have no significant effect on the strength of paper made from the fibers.

Past studies have indicated that the tensile strength of paper is not greatly influenced by fiber length (*13*). Jayme (*17*) found this to be true so long as the average fiber length exceeds 0.3 mm. Steinschneider and Grund (*18*) even demonstrated an increase in strength with decreasing fiber length on beating. This increase was probably due to better bonding.

In a recent study of the role of fiber strength in paper strength, Van den Akker, Lathrop, *et al.* (*19*) showed, with the use of dyed fibers, that the proportion of broken fibers in a rupture zone could be increased by increasing the bonding between fibers. The present work helps to illustrate the simultaneous influence of fiber strength and interfiber bonding strength on the tensile strength of paper in a specific system.

EXPERIMENTAL

This work involved a study of selected chemical changes in a pulp in an effort to show how they influence the tensile strength of paper made from the pulp. Two objectives were to be served. First, it was proposed to explain the tensile strength increase in chlorinated and extracted NSSC aspen pulp when it is bleached with hypochlorite. The second objective was to use the controlled conditions of this system to help elucidate, at least qualitatively, the contributions to tensile strength of fiber strength and capacity for fiber bonding.

Two aspen (*P. tremuloides*) which were 42 and 43 years old were felled, and the lower 2-ft. section of each tree was discarded in order to avoid reaction wood. The trees were sawed into logs, barked by hand, removing knots and limbs as best possible, and chipped in a Carthage two-knife chipper set to cut a $^3/_4$-in. chip. The chips were screened on a 4-mesh screen, and large pieces were removed by hand.

The wood was cooked in an A. O. Smith rotary digester with a water : wood ratio of 5 cc. water per g. of oven-dry wood. The cooking liquor contained 18.0% Na_2SO_3 and 5.0% Na_2CO_3 (based on the oven-dry wood). The initial pH of the liquor was 11.0 and the final pH was 8.8. The cooking schedule included a rise to maximum temperature of 172°C., over a 3-hr. period, and cooking at that temperature for an additional 1.5 hr. at a maximum pressure of 130 p.s.i. The charge was blown to a blowpit and immediately recovered for refining in a Bauer refiner with plates set at zero clearance, producing a fairly well-refined pulp with a minimum of shives.

The pulp was washed at high consistency and then screened on a 0.010-in. flat screen. It was dewatered by centrifuging, broken in a laboratory pulp shredder, and stored in polyethylene bags at 5°C., with formalin added as preservative. The yield was 66.0% and the permanganate number (TAPPI Routine Control RC-242) was 74, indicating a bleach demand of about 18.5% Cl.

Ten batches of the screened pulp were chlorinated in a laboratory chlorinator at a consistency of 2.55% with chlorine applied in the amount of 14.7% of the oven-dry pulp. The reaction was carried to exhaustion, and the pulp was washed with filtered tap water to a pH of at least 7.0. Extraction was carried out for 2 hr. at 40°C., in a Pfaudler mixer with 2% NaOH (based on the oven-dry pulp furnished to the chlorinator) at a consistency of 10%. Each extraction batch was washed thoroughly with warm filtered tap water and combined with the rest of the extracted pulp in a large stainless steel wash tank where the entire batch was washed with tap water for about 4 hr. The composite of all 10 batches is referred to as bleach 10. This was the starting material for the hypochlorite study.

The third bleaching stage was applied to the pulp immediately after the alkaline extraction to obviate the necessity for solvent drying prior to hypochlorite treatment. Jappe (*1*) found that storing the chlorinated and extracted pulp in the wet state caused a change in bleach demand. This phenomenon apparently is due to diffu-

Table I. Hypochlorite Treatment

Bleach	*Hypochlorite applied, % available chlorine*
10	0
11	0.5
12	1.5
13	3.0
14	6.0
15	9.0

sion of hypochlorite-consuming materials from the inner parts of the fiber structure to the surface, where they are immediately available to the hypochlorite. Solvent drying of the chlorinated and extracted pulp checked this tendency to change in bleach demand, but the solvent exchange procedure was difficult to duplicate.

Portions of the pulp from bleach 10 were treated with sodium hypochlorite solution ("Hi-lex"). Five different pulps were produced as indicated in Table I. The bleaching containers were 5-gal. earthenware crocks in which the pulp was stirred by hand. The conditions were: consistency of 10% oven-dry, temperature 40°C., pH maintained between 10.0 and 10.5 with caustic soda, and reaction time of 2 hr. or until the chlorine was exhausted. When the chemical was not exhausted within 2 hr., the reaction was stopped by the addition of sodium sulphite solution. The pulps were washed with hot, filtered tap water for about 20 min., then soaked for 4 hr. in the crocks, and finally thoroughly washed on a muslin-covered washbox. They were then dewatered to about 25% consistency, broken up in a laboratory pulp shredder, and stored in polyethylene bags at 5°C., with formalin added as preservative.

Handsheets for physical testing were made from unbeaten stock.

EXPERIMENTAL DATA

The experimental results are summarized in Table II.

Difficulties were encountered in the Klason lignin determination similar to those met by others (*1*). Several modifications of the Klason lignin method were attempted with little success. The procedure finally adopted included removing water from the pulp by successive extractions with alcohol and benzene. The pulp was then ground through a 20-mesh screen in a micro Wiley mill and treated according to TAPPI Standard T 222 m-54. Filtration of the lignin residue was accom-

Table II. Summary of Experimental Test Results

	Bleach					
	10	*11*	*12*	*13*	*14*	*15*
Chlorine applied (as hypochlorite), %	0	0.5	1.5	3.0	6.0	9.0
Pulp yield (oven-dry wood basis), %	54.9	53.4	53.3	52.5	51.5	51.1
G.E. brightness, pt.	51.8	66.2	80.4	83.4	85.4	86.2
Analytical Data						
Alcohol soluble (oven-dry pulp basis), %	1.12	1.22	1.36	1.17	1.32	1.20
Lignin, %[a]						
Klason	1.94	1.55	1.49	1.29	0.58	0.72
Apparent soluble	1.21	1.08	0.97	1.13	1.01	0.64
Total	3.15	2.63	2.46	2.42	1.59	1.36
Carbohydrates (oven-dry pulp basis), %						
5% hemicellulose (ash free)	13.8	14.1	14.0	14.5	14.6	14.4
16% hemicellulose (ash free)	5.8	6.1	6.3	6.6	5.4	5.5
Alkali-resistant cellulose (ARC)	78.1	77.7	77.0	76.1	76.6	76.3
Quality of carbohydrates:						
D.P. of 5% hemicellulose	130	150	150	140	135	125
D.P. of 16% hemicellulose	145	145	140	120	150	140
Weighted average D.P. of hemicellulose	135	145	140	135	145	135
Pulp viscosity, cp.	46.07	42.93	34.45	28.01	19.62	13.30
Approximate pulp D.P.[b]	1,990	1,940	1,810	1,680	1,460	1,220
Fiber Properties						
Hydrodynamic specific surface, sq. cm./g.[k]	15,390	15,330	...	15,780	16,340	16,650
Specific volume, cc./g.[k]	1.57	1.61	...	1.72	1.73	1.75
Water-retention value (WRV), %	320.1	315.1	321.5	322.7	324.8	325.9
Specific volume (calculated from WRV), cc./g.[c]	3.8	3.8	3.8	3.8	3.9	3.9
Weighted average fiber length, mm.[d]	1.03	0.92	...	0.93	...	0.87
Tracheids, %	87	79	...	87	...	89
Vessels, %	8	18	...	10	...	8
Screen classification of handsheet stock:						
On 20-mesh, %	9.1	9.7	8.5	7.6	4.7	0.5
On 20, through 35-mesh, %	45.3	47.8	48.1	48.0	48.9	46.7
On 35, through 65-mesh, %	26.7	25.1	25.8	26.0	26.5	28.9
On 65, through 150-mesh, %	12.6	12.4	12.7	12.8	13.1	14.9
Through 150-mesh (by diff.), %	6.3	4.3	4.9	5.6	6.8	9.0
Physical Properties of Handsheets[e]						
Thwing formation, units	46.3	47.8	50.0	48.0	50.8	59.2
Apparent density, g./cc.	0.677	0.676	0.704	0.734	0.745	0.782
Relative bonded area (RBA), %[k]	31.5	32.4	33.7	35.1	38.5	41.7
Viscosity-velocity product (VVP), kp. cm./sec.	3.04	4.10	4.53	5.50	5.47	6.42
VVP/RBA, kp. cm./sec.	9.65	12.65	13.44	15.68	14.22	15.41
Intrinsic fiber strength (zero-span breaking length), m.	13,550	13,100	13,160	13,050	11,620	11,100
Tensile breaking length, m.	4,325	4,490	5,160	5,160	5,340	5,800
Elongation, %[f]	2.01	2.05	2.39	2.43	2.32	2.46
Rupture work in tension, in.-lb.[g]	0.385	0.385	0.523	0.600	0.573	0.625
Tear factor	0.779	0.770	0.724	0.716	0.649	0.590
Breaking length, % on fiber strength	31.9	34.3	39.2	39.6	46.0	52.2
Increase in tensile strength, %[h,i]	...	5.3	12.5	19.5	28.7	34.3
Increase in relative bonded area, %[h,i]	...	2.5	7.0	13.0	23.2	31.7
Increase in VVP, %[i]	...	23.3	49.2	71.0	95.7	106.5

a Based on oven-dry, extracted pulp. *b* Determined from a slide rule designed by the Buckeye Cellulose Corp. *c* Pycnometric specific volume of cellulose taken as 0.62 cc./g. *d* Optical projection method. *e* Basis weight 65.2 g./sq. m. *f* Specimen length, 2.93 in. *g* Work required to rupture specimen 2.93 in. × 15 mm. wide. *h* Based on bleach 10. *i* Data from smoothed curves. *k* Determinations were carried out with assistance.

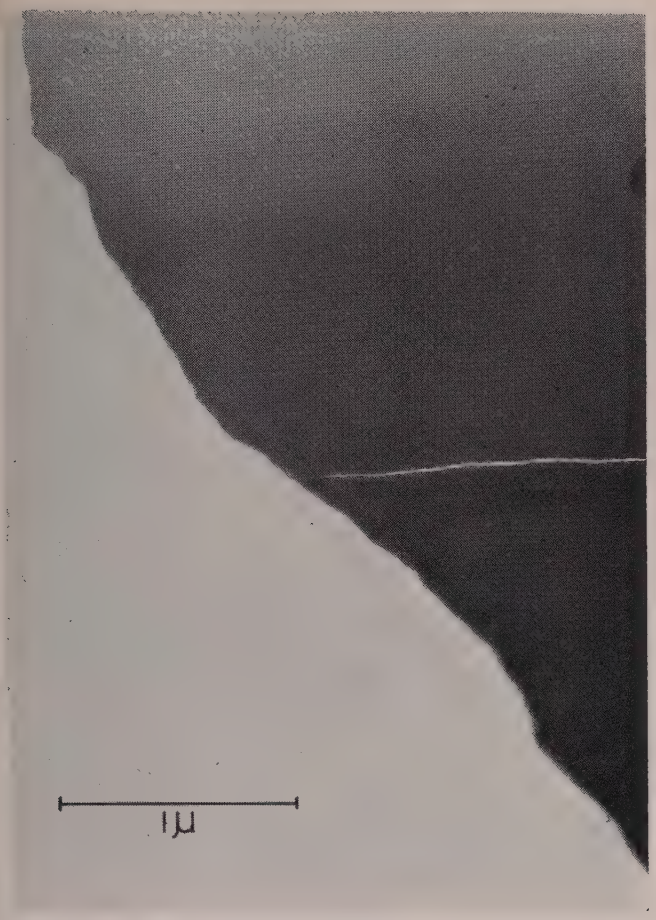

Fig. 1. Electron micrograph of typical bleach 11 fiber, showing smooth surface (chromium shadowed at 30°; light part is fiber)

plished on balanced Whatman no. 40 filter papers (*31*). The values reported are the averages of six determinations, and although there was some scatter in the data, the values reported are considered to be representative relative estimates of the actual lignin contents of the pulps.

When an attempt was made to obtain a summation of the analytical data in Table II, certain limitations became apparent. In some of the pulps, a summation of extractives, total lignin, and total carbohydrates gave a value over 100%; exceptions were found in the last two. There appeared to be an overlap of data—part of which might be due to the questionable so-called "soluble lignin." The data indicated no significant variation in the proportions of carbohydrates in the pulps.

The hydrodynamic specific surface area and effective specific volume were determined from constant rate filtration studies using the method of Ingmanson (*20*).

The data show that there was no difference in fiber length determined by the optical projection technique of Graff (*21*) but a screen classification and microscopic observation revealed that the bleached pulps had been deshived and had more fines. The fines consisted of parenchyma cells apparently released from fiber tracheids when the pulp was delignified by bleaching.

Since the examination of fibers in the light microscope showed no great differences between pulps, the electron microscope was used to examine two of the pulps. Water was displaced from the fibers, and they were mounted on grids and dried from butanol to preclude the tendency of fibrillae to bond back to parent fibers. Very significant differences then became evident between fibers from bleach 11 and bleach 15. Bleach 11 fibers could be characterized as intact, smooth-surfaced entities illustrated by Figs. 1 and 2. On the other hand, fibers from bleach 15 had many breaks in the surface with the outer wall wrinkled, checked, cracked, and severed, exposing the inner part of the fiber. The initial stages of this action were evidenced by a wrinkling of the surface. More progressive action resulted in the rupture of the outer wall as shown in Fig. 3. This layer appeared to be unfolded from the fiber and the free edge was generally curled in the direction in which it originally lay around the fiber. Figures. 1 to 4 illustrates part of an extensive area of the exposed wall material.

There was no significant change in the formation of the handsheets, within the reliability of the test, and the effect was not considered further.

The determination of relative bonded area was based on the method of Parsons (*22*) as modified by Ingmanson and Thode (*23*). The viscosity-velocity product (VVP) (*24*) was used as a measure of interfiber bonding strength. Fiber strength was measured by the zero-span tensile test. Van den Akker, Lathrop, *et al.* (*19*), using zero-span jaws of special design, have demonstrated a very good correlation between zero-span tensile strength and individual fiber strength for both coniferous and deciduous fibers. The tensile properties of handsheets were determined with a Baldwin-Southwark universal testing machine operating at constant rate of strain. Rupture work in tension was measured as the area under the load-elongation curve and represents a

Fig. 2. Electron micrograph of bleach 11 fiber showing intact fiber wall (chromium shadowed at 30°; light portion is fiber)

relative measure of the energy absorbed by a specimen before rupture.

DISCUSSION

The principal chemical changes noted were a reduction in the lignin and in the degree of polymerization of the alkali-resistant carbohydrates. The physical effects of the bleaching agent may be classified under two categories—enhancement of interfiber bonding strength and reduction of fiber strength.

Interfiber Bonding

The increase in hydrodynamic specific surface area from 15,300 sq. cm. per g. to 16,650 sq. cm. per g. can be attributed to deshiving, fibrillation, and swelling. A direct result of bleaching was a modest increase in the fines content of the pulp. This was demonstrated by the screen classification data. Microscopic observation of the fines fractions (through 150-mesh) of the various pulps, showed that they consisted almost entirely of parenchyma cells. With the removal of intercellular lignin, these fragments were free to exist as individual entities, whereas in the unbleached pulp they remained attached to the fiber tracheids.

The electron photomicrographs of Figs. 1 to 4 show a destructive effect on the outer wall of fiber tracheids with a consequent fibrillation. The hypochlorite treatment caused a wrinkling, cracking, and severing of the outer wall from parent fiber tracheids. The outer layer of fibers probably consists of a more or less coherent sheath composed of an interpenetrating system of carbohydrate and residual lignin. As a result of hypochlorite treatment, most of the lignin was removed, leaving a weaker structure so that a very mild swelling action caused it to wrinkle and rupture. Thus, as the lignin was removed from the outer regions of the fiber, fibrils were released to produce the fibrillated structure shown by Figs. 3 and 4. The surface so created was probably composed of cellulose and hemicellulose relatively free of lignin. It should be pointed out that this line of reasoning does not assume the chemical removal of a sheath as an entity, but rather the dissolution of part of the components in the layer.

Fig. 3. Electron micrograph of bleach 15 fiber showing outer layer of fiber wall split from parent fiber (chromium shadowed at 30°; light portion is fiber)

Fig. 4. Electron micrograph of bleach 15 fiber showing part of a detached outer layer held to the fiber by several fibrils (chromium shadowed at 30°; light portion is fiber)

Effective specific volume, as measured by the filtration technique, increased about 11% over the range of pulps studied. Although the change is only slightly greater than the accuracy of the determination, the upward trend is consistent. The water-retention value determinations (*25*) also indicate an increase in specific volume. This conclusion is based on the trend rather than on the magnitude of the change. Specific volumes calculated from water-retention values are much greater than seems reasonable. Specific volume is a measure of the volume of fiber plus water held in the fiber structure and thus is an indication of the extent of swelling in a pulp. Apart from the fact that the outer wall of a swollen fiber might be broken, it is probable that intrafiber bonding between fiber elements would be decreased so that internal shearing resistance to deformation would be reduced. The wetting, softening, and loosening action of a swelling agent would thus make the fibers more conformable.

Fiber conformability connotes a minimum resistance

Table III. Ratio of Relative Bonded Area to Hydrodynamic Specific Surface Area
(Based on data from smoothed curves)

Bleach	*RBA/S₀*
10	2.06×10^{-3}
11	2.11×10^{-3}
12	2.15×10^{-3}
13	2.25×10^{-3}
14	2.37×10^{-3}
15	2.50×10^{-3}

to deformation by mechanical or hydraulic pressures so that existing surfaces might be brought into such close proximity that hydrogen bonds may be formed at the critical stage of paper strength development.

The outer walls of fibers from the lightly bleached pulps appeared relatively smooth and probably consisted of an interpenetrating system of carbohydrate and lignin. Much of the area may have been shielded from bonding by lignin. In the case of those fibers with cracked surfaces, the secondary wall of the fiber was exposed. This possibly was carbohydrate relatively free of lignin and would be expected to be more apt to bond so that the portion of area bonded (relative bonded area) might be increased and so might the strength of bonding per unit of bonded area. The relative bonded area did, in fact, exhibit a continued increase over the range of pulps studied. Better fiber conformability might have permitted surface tension forces, at the critical stage of strength development, to deform fibers and

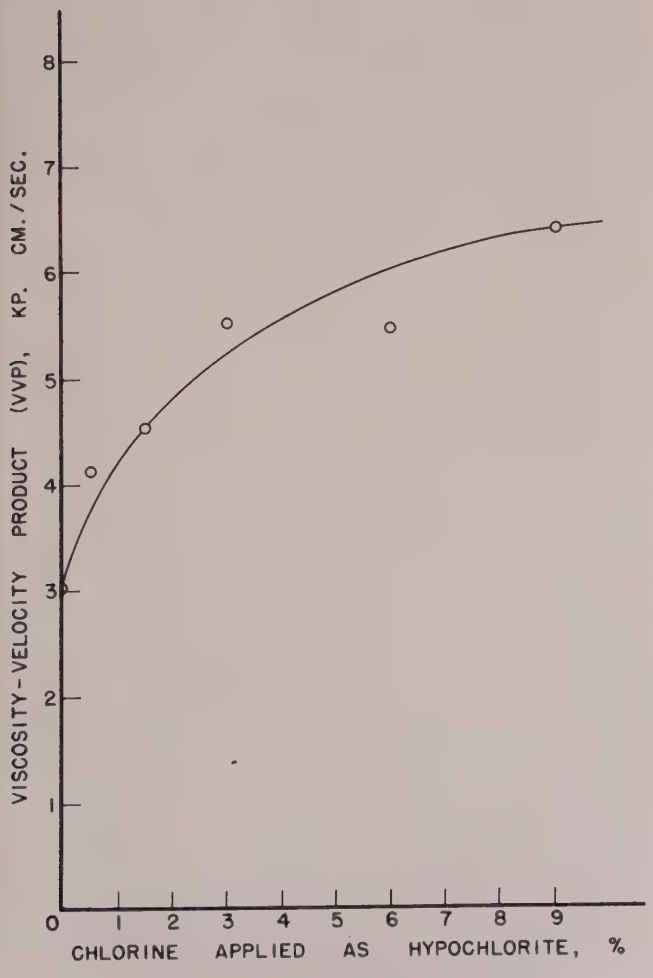

Fig. 5. Interfiber bonding strength

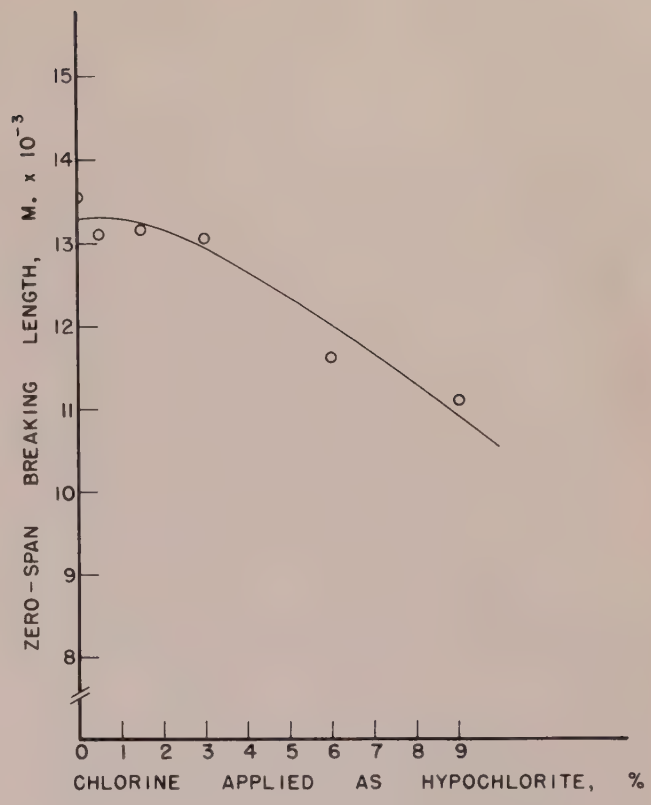

Fig. 6. Fiber strength

bring a greater proportion of existing surface into contact, increasing the extent to which this surface is utilized. Then, as a consequence of the existence of surface more apt to bond, a greater proportion did, in fact, bond together. The increased effectiveness of the original wet surface in achieving bonding in the final dried sheet is indicated by the ratios of relative bonded area to specific surface area tabulated in Table III.

The direct result of higher relative bonded area, and perhaps of greater bond density, was an increase in over-all interfiber bonding strength shown in Fig. 5. The increase in over-all bonding strength was greater than the increase in relative bonded area as demonstrated by the ratios of VVP to RBA in Table II. This continued increase in interfiber bonding strength per unit of relative bonded area must have been due either to an increase in bond density (more bonds per unit of bonded area) or to an increase in the strength of the bonds, or both. It is impossible, with present techniques, to separate these two possibilities. In the present case it is probable that both phenomena are involved. In the first place, it is reasonable to expect a greater bond density in the case of a pulp with lignin or other interfering constituents removed. Secondly, greater bond strength might be a legitimate speculation. Jappe's analytical data (*1*) indicated an increase in carboxylation of the cellulose resulting from hypochlorite treatment. Pauling (*26*) showed that hydrogen bridges involving carboxyl groups may have a greater bond energy than those in which aliphatic hydroxyl groups are partners.

Fiber Strength

Probably the only chemical change which had a direct bearing on fiber strength was a change in the D.P. of the cellulose. The viscosities of 0.5% cupriethylenediamine solutions of the pulps decreased consistently over

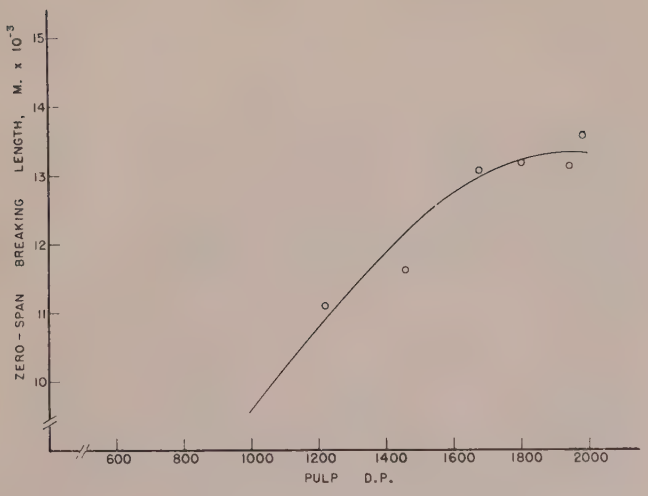

Fig. 7. Fiber strength—D.P. correlation

the range studied, and when converted to approximate D.P., indicate a loss from about 2000 to about 1200. An attempt to determine the D.P. of the alkali-resistant cellulose (A.R.C.) by measuring solution viscosity was unsuccessful because of excessive scatter in the data. Two possible explanations for this are suggested. There may have been extensive uncontrolled degradation of the cellulose, despite the precautions observed, during the alkaline preparation of the A.R.C. from bleached pulps. Also, uronic carboxyls in cellulose may form high molecular weight metal complexes in copper containing cellulose solvents. It was found that the D.P. of the hemicellulose fraction was not altered significantly by bleaching, and therefore it appears that the change noted for the pulp must be attributed to the A.R.C.

The effect of hypochlorite bleaching on fiber strength is depicted in Fig. 6. The application of a moderate amount of hypochlorite apparently did not affect the strength of the fibers much, even though the D.P. of the cellulose was altered. Overbleaching, on the other hand, caused a marked reduction in fiber strength.

A correlation between fiber strength and D.P. of the pup is shown in Fig. 7. Relationships of this nature have been presented for textile fibers (*16*, *27*, *28*), but there appears to be no such previous report for wood pulp cellulose. There is some question about a cause-and-effect relationship between fiber strength and molecular chain length. Past work (*27*, *28*) suggests a linear relationship between D.P. and fiber or film tensile strength over a relatively narrow range of low D.P. for regenerated cellulose products. However, as Mark (*29*) pointed out, increased D.P. above a certain minimum of about 500 appears to have no effect on the strength of regenerated fibers and films. On the other hand, evidence (*16*, *28*) has been presented in favor of such a correlation for natural fibers having D.P.'s up to about 10,000, both in the natural state and degraded by various agents.

Sheet Strength

A tensile specimen of paper under load is a structure embodying numerous entities held together by secondary valence forces. The load at which the specimen fails will depend on both the internal strength of the components and on the strength of the binding forces, with a limit imposed by the weakest link in the structure. The strength of a sheet of paper, at a particular level of bleaching, will therefore depend largely on the balance between fiber strength and interfiber bonding. The variation of handsheet breaking length with bleaching, for the pulps of this study, is shown in Fig. 8, and the trend may be explained on the basis of findings discussed above.

The progressive increase in interfiber bonding strength, through the series of pulps, had the effect of increasing sheet tensile strength. At the same time, although fiber strength was not changed much through the light bleaching treatments, it decreased considerably as the pulp was more heavily bleached. Thus, in the first part of the range studied, fiber strength had little effect because it was the strong link in the chain. In the latter part of the range, however, fiber strength was reduced to the extent that sheet strength was impaired.

The effectiveness of bonded area for achieving sheet tensile strength is further illustrated by Fig. 9. Ingmanson and Thode (*23*) found that, for a given pulp, the total surface area of water-dried fibers remains constant, and is independent of the degree of refining. Although swelling, fibrillation, and fines act to increase the surface area of wet pulp, the surface area of water-dried fibers remains the same, because fibrils and fines apparently bond to the surface of fibers and the effect of swelling is reversed by drying the pulp. Since the specific scattering coefficient of a pulp is a measure of scattering, or unbonded surface, the total bonded area in a sheet may be represented by the difference in scattering coefficients for water-dried, unbonded fibers and for the fibers in the sheet. Since the magnitude of the constant involved in the relationship between scattering coefficients and bonded area is not known, no attempt has been made to estimate the bonded area, but the difference in scatter-

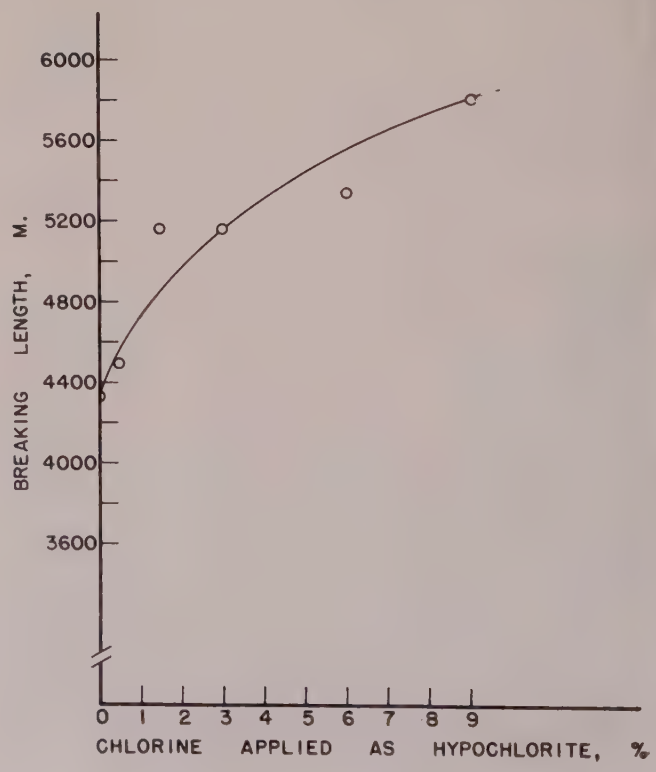

Fig. 8. Tensile breaking length

ing coefficients is taken as a number proportional to the total bonded area. Figure 9 shows the tensile strength developed by a given total "bonded area" for each of the pulps. Initially, the tensile strength increased very rapidly with bonded area, but as the bonded area became greater, the rate of change of tensile strength was reduced. Since there is no reason to expect a decrease in bonding strength per unit of bonded area for any of the pulps, the decreased slope of the curves is explained on the basis of increased influence of fiber strength. Thus, these curves illustrate the continually changing balance between interfiber bonding and fiber strength in defining the tensile strength of a sheet of paper. Figure 9 also illustrates the increased bonding strength per unit of bonded area of the pulps as the bleaching treatment was intensified. At any value of total bonded area, the unbeaten tensile strength of successive pulps was increased with greater bleaching. Since fiber strength was progressively reduced through the series, as shown by Fig. 6, the increased tensile strength must have been due to greater bonding strength per unit of total bonded area.

The concomitant variations in fiber strength, bonding and handsheet breaking length are reproduced in Fig. 10. It may be seen from this, and the data of Table II, that over-all bonding strength more than doubled over the range of the study, while handsheet breaking length increased only about 35%. It is interesting to note that fiber strength had sufficient influence, in the latter part of the range studied, that sheet strength was considerably impaired.

The decrease in tearing strength with bleaching, shown in Table II, supports the preceding explanation of the manner in which fiber strength and interfiber bonding influence paper strength. According to the modern concept of the mechanism of failure in the Elmendorf tear tester (*30*) the tearing strength depends on the balance between bonding and fiber strength. As the extent of bonding increases, relatively more fibers

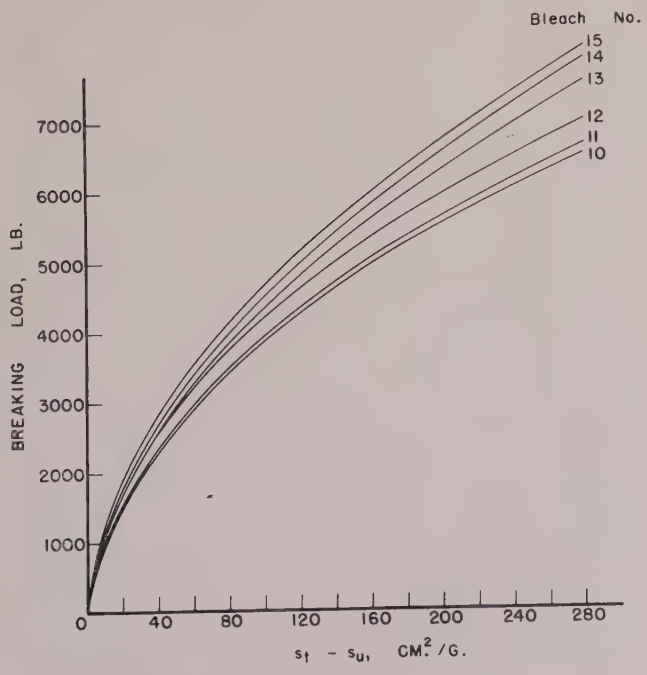

Fig. 9. Effectiveness of "bonded area" for achieving sheet strength

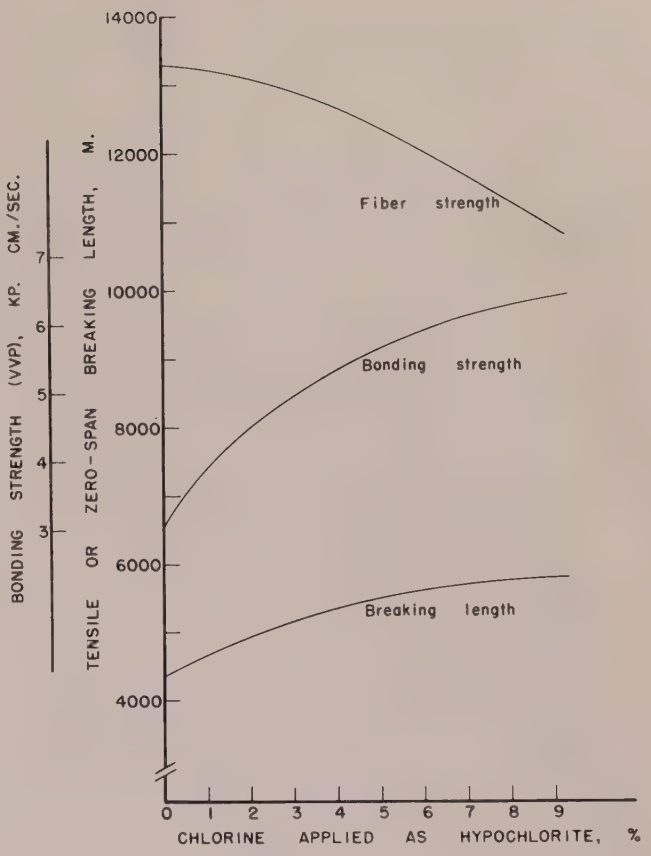

Fig. 10. Concomitant strength changes

are broken and fewer are pulled from the sheet structure. Since shorter distances are involved in breaking fibers, less work is required to tear the sheet.

CONCLUSIONS

The major chemical changes when chlorinated and extracted NSSC aspen pulp is treated with hypochlorite are delignification and a reduction in the D.P. of the pulp. Swelling, deshiving, and fibrillation result in an increase in hydrodynamic specific surface area. Handsheets made from the bleached pulp had more bonded area and increased bonding strength per unit of bonded area. The increase in bonding strength was greater than the increases in surface area and bonded area, suggesting that bleaching provided surfaces better capable of bonding.

Bleaching was also responsible for concomitant decreases in fiber strength and in the D.P. of the pulp. A correlation has been shown between fiber strength and pulp D.P. It is possible that this is a forced correlation because the loss in fiber strength might be attributed in part to other causes. The maximum zero-span breaking length of handsheets of this study was 13,300 meters, indicating a zero-span fiber strength of 21 kg. per sq. mm. A statistical treatment of fiber distribution in handsheets (*19*) shows that the zero-span breaking length should be 37.5% of actual fiber strength. On this basis, the maximum fiber strength was about 56 kg. per sq. mm.

Hypochlorite bleaching of the pulp was responsible for increased breaking length of handsheets prepared from the unbeaten pulp. This strength increase was due to increased interfiber bonding strength. Sheet

tensile strength was limited at high levels of bleach application because of the adverse effect on fiber strength.

The tensile strength of handsheets in this study was found to depend on both fiber strength and interfiber bonding. If other factors are equal, the level of strength will be determined by the *balance* between fiber strength and bonding but limited by one or the other. These observations support and broaden the concept of balanced fiber strength and fiber bonding in establishing the strength of paper.

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The Hypothesized Carbonic Acid Ester Linkages in Cellulose Oxidized by Aqueous Chlorine at pH 4.5

JULIAN W. DANIEL

A series of acid (pH 4.5) chlorine-oxidized celluloses was prepared from bacterial cellulose-1-C^{14}. Each of these at a consistency of 5% was treated with 0.025 *N* NaOH for 12 hr., the slurry was subsequently acidified with normal sulphuric acid to liberate carbon dioxide. On the basis of the specific activity of the saponifiable carbon dioxide (SCD), not more and probably much less than 20% of it was derived from position 1. In view of this, the probability of the existence of the carbonic acid ester linkage in this type of oxidized cellulose is greatly reduced; therefore, the contribution of such a linkage to the alkali liability of acid chlorine-oxidized celluloses is minor. Of the "uronic acid" carbon dioxide evolved from oxidized and unoxidized cellulose-1-C^{14} and cellulose-6-C^{14}, only a small portion came from position 6; consequently, such results are meaningless when interpreted in terms of uronic anhydride. The specific activity of the "uronic acid" carbon dioxide from cellulose-1-C^{14} decreased as the level of oxidation and the per cent of "uronic acid" carbon dioxide increased. This indicates a change in the fraction of the carbon dioxide coming from the various positions in the cellulose as a function of oxidation level. A comparison of physical and chemical properties of cotton and bacterial celluloses, bath oxidized and unoxidized, indicated that these two celluloses could be used interchangeably.

The action of aqueous chlorine on cellulose has been widely investigated (*1–7*). Apparently, chlorine, hypochlorous acid, and the hypochlorite ion are nonspecific oxidizing agents, which react with the cellulose macromolecule at different points and lead either to the "reducing" type of cellulose or to the "acidic" type, as defined by Birtwell, Clibbens, and Ridge (*1*). The most recent outstanding study dealing with the chemical changes produced in cellulose by chlorine, hypochlorous acid, and the hypochlorite ion was made by the Soviet workers, Kaverzneva, Ivanov, and Salova (*4–7*). This work represents one of the most detailed chemical (functional group) analyses ever made on a series of oxidized celluloses. Their data led to numerous statements on the nature of these celluloses. Further study of one of their proposals, the hypothesis that carbonic acid ester linkages (see Fig. 1) are present in cellulose oxidized by aqueous chlorine at pH 4 to 5* [predominantly aqueous hypochlorous acid (*8*)], is the main subject of this paper.

The possibility of carbonic acid ester linkages in defective cellulose was first suggested by Staudinger (*9, 10*); however, no experimental evidence was presented for the support of such a structure. Kaverzneva, *et al.* (*4–7*), revived this hypothesis on the basis of

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* Aqueous chlorine at pH 4.3 ± 0.3 is the only oxidative system of interest in this research; therefore, oxidized celluloses will henceforth be denoted either acid chlorine-oxidized cellulose or simply oxidized cellulose.

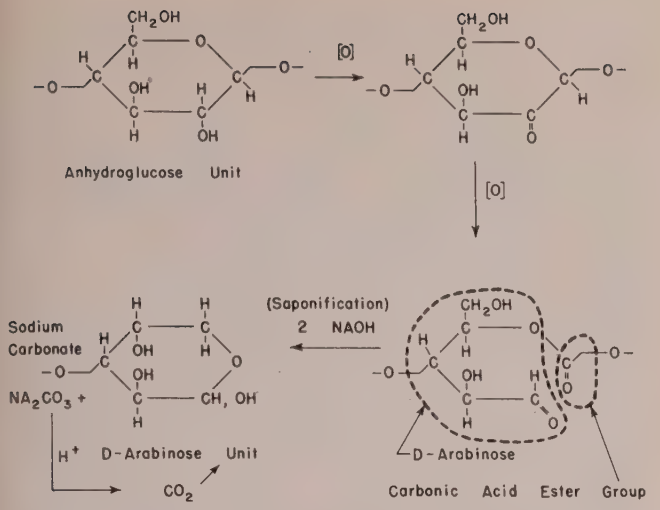

Fig. 1. Formation of hypothesized carbonic acid ester linkage, according to Kaverzneva (4)

suggestive, but inconclusive, evidence; thus the primary aim of the research discussed below was to test the validity of the carbonic acid ester hypothesis. The merit of this undertaking stems from the role that such a structure might play in the alkali sensitivity of acid chlorine-oxidized cellulose (*11*).

Kaverzneva, *et al.*, oxidized cotton with aqueous chlorine (buffered at about pH 4.5 with an acetate buffer system), then washed, dried, and saponified it with a 0.025 *N* solution of sodium hydroxide.† After an interval of time, the saponification mixture was acidified and carbon dioxide‡ was liberated. This carbon dioxide was ascribed to the saponification of carbonic acid ester linkages. Unoxidized cellulose and cellulose oxidized in an alkaline medium gave off almost no carbon dioxide.

Meller (*13*) discussed the data presented by Kaverzneva, *et al.*, and concluded that the hypothesized carbonic acid ester linkage was consistent with the existing information. However, he noted that further evidence was needed in order to verify such a hypothesis.

Henderson (*14*) reported the isolation and characterization of D-arabinose from cellulose oxidized by aqueous chlorine at pH 4.5. He stated that the D-arabinose was derived from an oxidative cleavage of carbons 1 and 2 of the glucopyranose unit. The carbon 2 was left as a carbonyl group, and this became the carbon 1 of the arabinose unit. The original carbon 1 of the glucopyranose unit was left as a carbonic acid ester hypothesis advanced by Kaverzneva, *et al.*

Kaverzneva, *et al.* (*5*), have used the Lefévre and Tollens (*15*) uronic acid method in the interpretation of very minor differences in the nature of acid chlorine-oxidized celluloses. These interpretations have been made in light of evidence which prompted Browning (*16*) to state that amounts of "uronic acid"§ carbon dioxide in the range of 0.2 to 0.4%, based on the cellulose, cannot be considered definite evidence of the presence of uronic acid groups.

† The cellulose ester of methyl carbonic acid is unstable in the presence of alkali (*12*).

‡ Carbon dioxide derived from oxidized cellulose by this procedure will subsequently be called saponifiable carbon dioxide (SCD).

§ Carbon dioxide liberated from cellulose by application of any adaptation of Lebévre and Tollens' uronic acid method.

MATERIALS

Purified Cotton

Ginned and cleaned cotton was obtained from the Southern Regional Laboratories of the Department of Agriculture at New Orleans, Louisiana. It was Wiley-milled through 1/4-in. holes, extracted for 3 hr. with absolute ethyl alcohol under reflux (1500 ml. per 105 g.), then washed with acetone and air-dried. It was next extracted for 2 hr. with 1% KOH under reflux (3500 ml. per 105 g.), filtered, washed with 1% CH_3COOH (100 ml. per 105 g.), and then washed with water until the washings were neutral. It was next washed with absolute ethyl alcohol and acetone (2 l. each per 105 g.), air-dried, and stored.

Labeled Bacterial Cellulose

Greathouse's (*17*) method for the preparation of labeled bacterial cellulose, with minor changes, was scaled up for the biosynthesis of about 30 g. of cellulose per batch; one hundred 3000-ml. Erlenmeyer flasks were used for the operation. The medium in each flask had about 1.7 sq. cm. of air-liquid interface per cc.

Acetobacter xylinum, A.T.C.C. strain no. 12733, was utilized for the growth of the bacterial cellulose. The nutrient medium used consisted of an aqueous solution of 0.5% glucose, 2% Difco yeast extract, and 0.1% KH_2PO_4 (weight-to-volume percentages, w/v). The initial pH of the medium was 6.0. Inoculation was made by transfer of a 3-day-old colony suspended in about 5 ml. of a 1-day-old culture of the bacterium in nutrient medium to each flask of sterile medium. The cellulose pellicles were collected from the flasks 7 days after inoculation, washed with hot tap water (250°C.) for about 8 hr., and then autoclaved for 2 hr. at 15 lb. p.s.i. gage in 1% NaOH (1500 ml. per 6 g. cellulose). They were next washed with water, then with a 0.5% CH_3COOH solution, and again with water until the washings were neutral. The cellulose was dried by solvent replacement, using absolute ethyl alcohol, acetone, and diethyl ether, in that order, then Wiley-milled past a 40-mesh screen, and stored.

For the preparation of carbon-14 labeled cellulose, 60% of the total glucose addition, including all of the labeled D-glucose, was added after the physiological activity of the bacterial population had passed its peak, about 24 hr. after inoculation. This delay in the addition of the labeled glucose gives a marked increase in activity yield and specificity‖ of label.

Special Solutions

Carbon dioxide-free water was prepared by bubbling nitrogen through triple distilled water at 95°C. for 30 min. The bottle containing the water was tightly stoppered, and as the water cooled, the pressure was allowed to equilibrate through an Ascarite (asbestos and sodium hydroxide) guard tube. The carbon dioxide-free water was siphoned off as needed.

Carbonate-free sodium hydroxide solutions were prepared from Acculutes¶ of concentrated sodium

‖ Specificity denotes the percentage of the total radioactivity present in a molecule that is present in a specific position.

¶ Acculute is the trademark of standard volume solution prepared by Anachemia Chemicals Ltd.

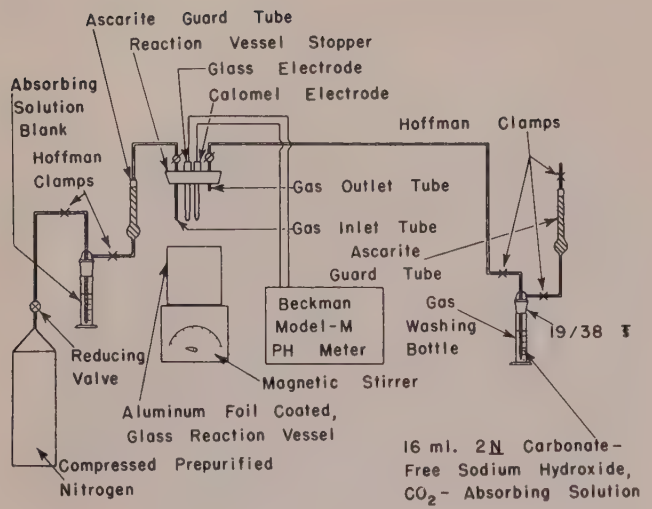

Fig. 2. Schematic of cellulose oxidation apparatus

hydroxide (carbonate-free) and carbon dioxide-free water. The preparation and transfer of these solutions were conducted in a carbon dioxide-free constant-atmosphere box with neoprene arm tubes, kept under a positive pressure with compressed air. This air was first passed through a calcium chloride column, an Ascarite column, and a soda-lime solution. The solutions were stored in polyethylene bottles.

APPARATUS AND METHODS

Cellulose Oxidation

All oxidations were performed with an aqueous chlorine system buffered at pH 4.5 with either an acetate or a phosphate buffer system.

The oxidation apparatus is schematically shown in Fig. 2. The reaction vessel used was a 400-ml. wide-mouth glass bottle, covered with aluminum foil. On initiation of an oxidation, the reaction vessel was sealed with a rubber stopper, fitted with a glass electrode, a calomel electrode, a gas exit tube, and a gas inlet tube. The inlet tube extended to near the bottom of the vessel, and the exit tube extended into the vessel but not into the reaction mixture. The electrodes were connected to a Beckman Model M pH meter. A magnetic stirrer was used for occasional agitation. Near the end of the oxidation, the gas inlet tube was opened to allow purified nitrogen to pass into the reaction mixture to purge it of dissolved carbon dioxide; the remainder of the time, the inlet tube was closed. The carbon dioxide evolved during the oxidation passed out of the reaction vessel and bubbled into a carbonate-free sodium hydroxide-absorbing solution. The bubbles were ejected from a 2-mm. diam. capillary at the bottom of the gas-washing bottle; to exit these bubbles passed through 16 ml. of 2 *N* caustic solution (45 mm. distance). Ascarite guard tubes were so placed in the apparatus that they protected the carbonate-free sodium hydroxide solutions from contamination.

The oxidant was prepared by bubbling chlorine through a normal solution of sodium hydroxide until the pH dropped to 9.5. After cooling and just prior to using, the hypochlorite solution was standardized according to White (*18*) with respect to chloride, available chlorine, and chlorate.

About 12 g. of cellulose were used for each oxidation; it was suspended in buffer solution with sufficient buffering capacity to hold the pH at 4.3 ± 0.3 throughout the oxidation. The pH of the hypochlorite solution was adjusted to 4.5 with either acetic or phosphoric acid (depending on the buffer system) immediately before addition of the oxidant to the reaction vessel containing the buffered cellulose slurry. The reaction mixture was made up to 5% consistency, and the vessel was sealed. The oxidation was allowed to proceed for 40 hr. at room temperature (25 ± 5°C.); at this point, all or nearly all of the oxidant had been consumed. On completion of the oxidation, the gas-washing bottles containing the carbon dioxide-absorbing solution and the absorbing solution blank were closed and set aside for analysis. The spent oxidizing solution was then separated from the oxidized cellulose; it was analyzed for chloride, available chlorine, and chlorate (*18*), and the oxidant consumption was calculated. The oxidized cellulose was washed with water, 1.5% $NaHSO_3$ solution, water, 0.5% acetic acid, water, absolute ethyl alcohol, acetone, and ether, in that order. The oxidized cellulose was vacuum-dried to constant weight (at 735 mm. Hg vac. and 50°C.) with the yield being determined; it was then stored in a tightly sealed weighing bottle.

Cellulose Saponification

About 2 g. of cellulose were suspended in carbon dioxide-free water. This mixture was then made 0.025*N* in sodium hydroxide by the addition of carbonate-free sodium hydroxide. This alkaline mixture was allowed to stand for 12 hr. before it was acidified (pH 3) with normal sulphuric acid. The carbon dioxide liberated by this treatment was determined either titrimetrically or manometrically; these methods gave comparable results.

Determination of Carbon Dioxide

Titrimetric Micromethod. The confidence limits (95%) for data obtained by this method were ±0.04 mg. of CO_2; the sample size utilized for this analysis was approximately 0.8 mg., as CO_2.

A brief description of the component parts of the apparatus (Fig. 3) follows: (1) prepurified nitrogen

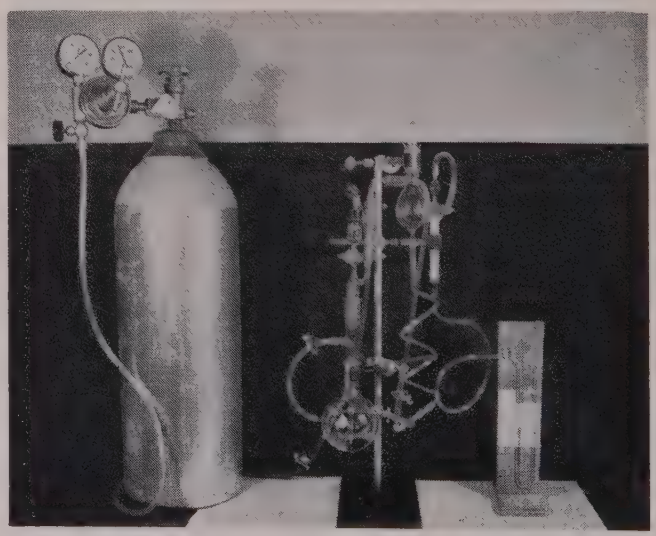

Fig. 3. Apparatus for the titrimetric determination of carbon dioxide

supply; (2) reaction flask with gas inlet tube extending to the bottom of the flask; (3) acid reservoir above reaction flask, 5-ml. volume; (4) Hoffman clamp; (5) gas inlet tip, 2-mm. capillary tube with 1-mm. opening into absorption tube; (6) absorption tube, 8-mm. glass tubing, 50 cm. in length; (7) stoppered absorption flask with a side arm; (8) stopcock, absorption flask side arm; (9) Ascarite guard tube, and (10) water manometer. Tygon tubing was used exclusively where tubing connections were made in apparatus.

The carbonate sample was placed in the reaction flask, 2, and the flask was connected to the apparatus. Ten milliliters of 0.02*N* $Ba(OH)_2$ hydroxide (standardized with 0.01*N* HCl before each run) were added to the absorption system through the top of the absorption flask; the flask was then quickly closed. A flow of nitrogen was started through the apparatus at a flow rate of about 1500 ml. per hr. At time *zero*, adequate normal sulphuric acid was added to the reaction flask from the acid reservoir, 3, to decompose the carbonate solution to carbon dioxide. The flow of nitrogen was continued for 3 hr. After completion of the run, the absorption system was closed at points 4 and 8 and was severed from the apparatus. The barium hydroxide-absorbing solution was washed from the absorption system with carbon dioxide-free water under nitrogen pressure. The washings were removed through the side arm of the absorption flask and were collected in a 125-ml. flask covered with dental dam; the total washings amounted to 50 ml. Phenolphthalein was added to the solution; the solution was titrated through dental dam with 0.01 *N* HCl. The amount of carbon dioxide absorbed by the barium hydroxide solution was calculated; this result was corrected for a blank.

Manometric Micromethod. The carbon dioxide, liberated from a carbonate solution, was manometrically determined according to the Van Slyke-Folch method (*19*). The submicro technique was used for samples with 0.15 to 0.7 mg. of carbon, and the micromethod was used for samples with 0.7 to 3.0 mg. of carbon.

Uronic Acid Determination

The method used by Browning (*20*) with two modifications was used for analyzing celluloses for their uronic acid content; the modifications were: (1) A sodium hydroxide solution, instead of Ascarite, was used for the absorption of carbon dioxide. The carbon dioxide absorbed was determined by the manometric method. (2) The nitrogen flow rate through the apparatus was changed from 8 to 6 cc. per min.

The gas washing bottle used for the absorption of the carbon dioxide contained 16 ml. of 0.15 *N* carbonate-free NaOH. This absorption vessel was identical with that used in the determination of the carbon dioxide evolved during cellulose oxidation.

This uronic acid method was checked with known glucurone; it gave results that were about 7% below theory for samples yielding approximately 10 mg. of CO_2.

Characterization of Labeled Cellulose—Positions 1 and 6

The cellulose was hydrolyzed to glucose by the Forest Products method (*21*); the hydrolyzate was concentrated, and the glucose was oxidized to potassium gluconate by the method of Moore and Link (*22*). The specific activity** of the total carbon in the potassium gluconate was determined according to the wet combustion-gas phase counting method of Van Slyke, Steele, and Plazin (*23*).

A modified form of Eisenberg's (*24*) method was used for the determination of the specific activity of the carbon in positions 1 and 6 of the potassium gluconate; the modifications were: (1) the carbon dioxide from each position (*19*) was determined manometrically, and (2) the radioactivity of the carbon dioxide was determined by the gas phase proportional counting technique of Van Slyke, Steele, and Plazin (*23*). (The radioactivity of all carbon dioxide samples analyzed in this research was determined by the above-mentioned technique.)

Chromatographic Techniques

Descending paper chromatographic methods were used for the qualitative analysis of the neutral sugars in cellulose hydrolyzates. Piper and Bernardin's (*25*) spectrophotometric method was used for the quantitative determination of pentose and hexose sugars. A fractionation procedure utilizing Whatman 3-mm. paper was developed for the purification and concentration of certain trace fractions of the hydrolyzates. (Whatman no. 1 filter paper was used elsewhere.) A number of developers and spray reagents were used; the most useful of these were:

Developers: A. Butanol, pyridine, water (10:3:3); B. Ethyl acetate, acetic acid, formic acid, water (18:3:1:4); C. Methyl ethyl ketone saturated with water.

Spray Reagents: A. 2-Amino biphenyl (sugars) (*26*); B. *p*-Anisidine hydrochloride (sugars) (*27*); C. 1% Aqueous methyl orange (acids); D. Ninhydrin (amino acids) (*28*); E. Hydroxylamine (lactones) (*29*); F. Anthrone (ketoses) (*30*).

Cellulose hydrolyzates for chromatographic analysis were prepared as follows: One-half gram of cellulose was treated with 9 ml. of 72% sulphuric acid at room temperature (about 25°C.) for 1 hr. The mixture was then diluted with 207 ml. of distilled water and refluxed for 3 hr. The cooled solution was neutralized to pH 6.0 with barium hydroxide, filtered through a Celite-activated carbon bed, passed through an Amberlite IR-120 ion-exchange column, and concentrated to dryness *in vacuo*. The syrup was redissolved in a small quantity of water and filtered through glass wool. The solution was then made up to the desired volume.

Degree of Polymerization

The degree of polymerization (D.P.) of oxidized and unoxidized celluloses was determined by viscosity measurements. Solutions of cellulose as cellulose trinitrate (CTN) in acetone (nonalkaline) and cellulose in 0.5 *M* cupriethylenediamine (CED) (alkaline) were both used so that alkali lability could be assessed. The intrinsic viscosities $[\eta]$ for these solutions were determined and converted to D.P. using the relationship $[\eta] = K_w M_w^a$. The constants for CTN in acetone are K_w equals 1.7×10^{-5} and a equals 1.00; these were based on light-scattering measurements of Benoit and Doty (*31*). The constants for CED solutions were obtained by the direct comparison of intrinsic viscosity values for CED solutions of unoxidized celluloses and the acetone solutions of the trinitrates of these celluloses. This was

** The specific activity of a substance is the radioactivity per unit weight of the substance, e.g., c.p.m./mg., etc. (c.p.m. = counts per minute).

Table I. Cellulose Oxidation Data and SCD Results

Number	Sample identification (oxidation number, etc.)	Material	Buffer system (pH 4.3 ± 0.3)	Oxidant consumption,[d] equiv. HOCl/mole anhydroglucose[a]	Yield,[b] %	Method of SCDc determination	SCD,[d] mole CO_2/100 moles anhydroglucose
1	Oxidation I (Sample 1)	Cotton	Acetate	4.34	74.3	Titrimetric	0.37
2a	Oxidation II (Sample 2)	Cotton	Phosphate	2.59	80.3	Titrimetric	0.38
2b	Oxidation II (Sample 2)	Cotton	Phosphate	2.59	80.3	Manometric	0.35
3	Oxidation II (Sample 3)	Cotton	Phosphate	2.66	77.8	Titrimetric	0.37
4	Oxidation X	Cotton	Acetate	1.54	82.0	Manometric	0.25
5	Oxidation VI	Bacterial cellulose	Acetate	0.29	96.2	Manometric	0.10
6	Oxidation VII	Bacterial cellulose	Acetate	0.82	89.7	Manometric	0.15
7	Oxidation XII	Bacterial cellulose	Phosphate	0.91	89.8	Manometric	0.15
8	Oxidation VIII	Bacterial cellulose	Acetate	1.37	86.9	Manometric	0.17
9	Oxidation IX	Bacterial cellulose	Acetate	1.38	86.1	Manometric	0.18
10	Oxidation XI	Bacterial cellulose	Acetate	1.39	85.9	Manometric	0.19
11	Oxidation IV	Bacterial cellulose	Phosphate	2.31	77.8	Titrimetric	0.24
12a	Oxidation V	Bacterial cellulose	Acetate	2.65	84.1	Manometric	0.25
12b	Oxidation V	Bacterial cellulose	Acetate	2.65	84.1	Manometric (one hour)	0.11
13	Unox. purif. cotton	.....	.....	..	..	Titrimetric	0.02
14	Unox. purif. bact. cellu.	.....	.....	..	..	Titrimetric	0.01

[a] Purified cotton and bacterial celluloses are assumed to be 100% anhydroglucose, i.e, for calculations of this type.
[b] Yields are calculated on o.d. basis; o.d. is defined herein as a sample that has been dried over P_2O_5 at 30°C. and 29 in. Hg vacuum for 12 hr.
[c] SCD = saponifiable carbon dioxide.
[d] Averages of duplicate determinations.

done on the assumption that no alkali-labile linkages were present in the unoxidized cellulose; therefore, both methods would give rise to identical D.P. values. The CED constants were K_w equals 3.0×10^{-5} and a equals 1.00.

Cellulose nitrate was prepared according to the method of Bennett and Timell (*32*). The degree of nitration of the cellulose nitrates was determined by the semimicro Kjeldahl method of Timell and Purves (*33*). The technique of Browning, Sell, and Abel (*34*) was used for the preparation of 0.5*M* cupriethylenediamine solutions of cellulose. The viscosities were measured in Cannon-Fenske viscometers at 20°C.

RESULTS AND DISCUSSION

Chemical Studies

Saponifiable Carbon Dioxide in Oxidized Celluloses. SCD was determined on eleven different oxidized celluloses (Table I). The degree of oxidation of these celluloses varied from 0.3 to 4.3 equiv. of hypochlorous acid per mole of anhydroglucose. For the oxidized bacterial cellulose samples examined the quantity of SCD was less than that in oxidized cotton cellulose at comparable levels of oxidation (see Fig. 4).

From the results reported by Kaverzneva, *et al.* (*5*), it appears that the SCD in oxidized cellulose is related to the oxidant consumption; this is substantiated by results reported herein (see Fig. 6). For eight oxidized bacterial celluloses, the SCD increased for each significant increase in oxidant consumption; no definite leveling off point was reached at the highest level of oxidation attained. However, for samples of oxidized cotton, the SCD content leveled off at about 0.37 mole CO_2 per 100 moles of anhydroglucose, corresponding to an oxidant consumption of about 2.5 equiv. of hypochlorous acid per mole of anhydroglucose. Kaverzneva, *et al.* (*5*), suggested that this leveling-off effect was due to a balance between the formation and the destruction of the saponifiable groupings.

A 1-hr. saponification treatment was found to give rise to less SCD than did a 12-hr. treatment; after 12 hr., no additional increase was noted (see items 12a and 12b in Table I). This is felt to be due to the time aspect of the penetration of the 0.025 *N* NaOH solution into the cellulose and the subsequent alkaline decomposition of the carbon dioxide-yielding structures. The results reported by Kaverzneva (*4*) were derived from 1-hr. saponifications, whereas, unless otherwise noted, 12-hr. saponifications were used in this work. It will be shown later that the SCD from a 1-hr. saponification apparently has the same origin as that from a 12-hr. treatment.

Kaverzneva (*4*) stated that the carbonic acid ester linkage, her hypothesized source of all the SCD, is rather unstable; however, other than the action of dilute caustic (0.025 *N*), she gave no supporting evidence for this statement. In the present research, the SCD in an oxidized cotton cellulose (0.37 mole CO_2 per 100 moles anhydroglucose) was not observed to be

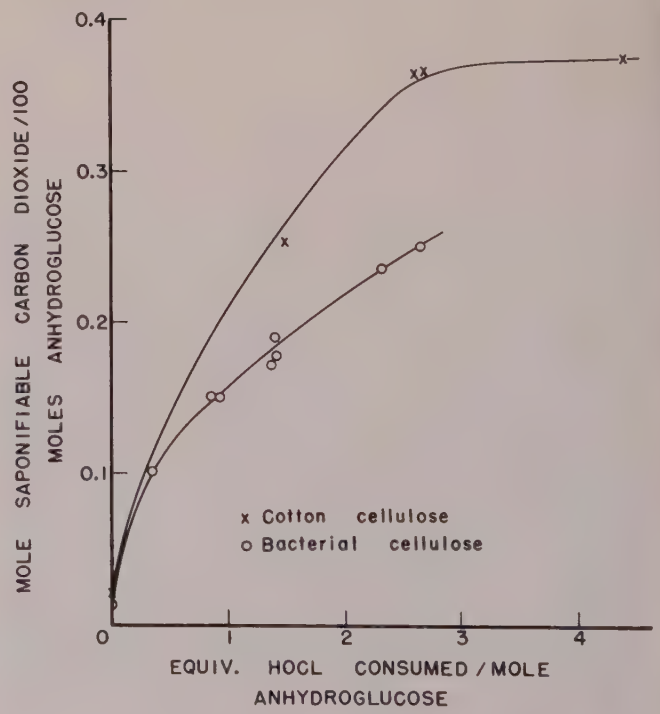

Fig. 4. Effect of oxidant consumption on saponifiable carbon dioxide content of oxidized cellulose

Table II. Purified Labeled Bacterial Cellulose Preparations

Purified labeled bacterial cellulose	Yield,[a] g.	Yield,[b] %	Yield of C^{14}-carbon, %	Nitrogen in cellulose, %	D.P. of cellulose	Sp. act. of anhydroglucose unit, c.p.m./mg. C[c]	Sp. act. in C(1)-position, c.p.m./mg. C	Activity[d] in C(1)-position, %	Sp. act. in C(6)-position, c.p.m./mg. C	Activity[e] in C(6)-position, %	Estimates[f] of sp. act. in C(2)-, C(3)-, C(4)-, and C(5)-positions, c.p.m./mg. C			
											C(2)	C(3)	C(4)	C(5)
Batch 1: Cellulose-1-C^{14}, used in oxidation V	36.3	40.2	16.1	..	..	2774[g]	13,313	80.2	..	..	..	..	..	..
Batch 2: Cellulose-1-C^{14}	36.0	40.0	17.8	..	..	3092	..	..	..	..	..	..	..	..
Batch 3: Mixture of batches 1 and 2, used in oxidations VI, VII, VIII, and XII	..	..	..	0.20	3370	2914	13,953	79.8	362	2.1	110	1950	1050	110
Batch 4: Cellulose-6-C^{14}, used in oxidation IX	31.7	35.2	21.3	0.07	2750	1920	2773	24.1	6807	59.1	450	1050	400	350

[a] For all calculations, cellulose assumed to be 100% anhydroglucose.
[b] Yield based on glucose supplied.
[c] Specific activity, counts per minute per milligram of carbon.
[d] Specificity at carbon 1.
[e] Specificity at carbon 6.
[f] Estimates calculated on basis of distributions of label in bacterial celluloses, as presented by Greathouse (*17*), and total activity of anhydroglucose units.
[g] All activity values represent average of two determinations, counting error less than 3% for all counts made in this research.

affected significantly by either an acidity of pH 3 (0.36) or a 15-min. treatment with distilled water at about 50°C. (0.35).

Neutral Oxidation Products in Oxidized Cellulose. Arabinose and erythrose were chromatographically identified as oxidation products in the hydrolyzates of oxidized cotton and bacterial celluloses. In addition, xylose was detected in the hydrolyzate of an oxidized bacterial cellulose. (None of the foregoing sugars was detected in the hydrolyzates of the unoxidized celluloses.) The xylose was found by taking two fractions from the pentose region of developed fractionation chromatograms; one corresponding to an arabinose known and the other to a xylose known. In the fractionation of the oxidized cotton hydrolyzates, only the fraction corresponding to arabinose was taken for analysis; thus, xylose may have been present although it was not detected. The identification of arabinose qualitatively confirmed the finding of Henderson (*14*). However, no more than 0.2% arabinose by weight (sample 2, oxidation II) could be found in the oxidized celluloses examined, whereas Henderson reported that 1% was present in his oxidized cellulose. This discrepancy remains unexplained. The $R_{glucose}$ values for arabinose, xylose, and erythrose in developer A (butanol, pyridine, water—10:3:3) were 1.32, 1.67, and 3.14, respectively.

The suggested origins of the chromatographically identified neutral oxidation products follow: (a) Arabinose may be derived from end-group oxidation and/or the oxidative cleavage of the bond between carbons 1 and 2 of the anhydroglucose unit, leaving carbon-2 as an aldehyde group. (b) Erythrose may be formed by the oxidative cleavage of the C(2)-C(3) glycol grouping of the anhydroglucose unit and/or further oxidation of the arabinose units. (c) Xylose may be formed by the decarboxylation of glucuronic acid groups occurring during either the oxidation or the hydrolysis of the oxidized cellulose.

Ratio of Saponifiable Carbon Dioxide to Arabinose in Oxidized Cellulose. Henderson (*14*) stated that the oxidative cleavage of anhydroglucose units between carbons 1 and 2 during acid chlorine oxidation of cellulose gives rise to D-arabinose and carbonic acid ester groupings at the C(1)-position in the oxidized cellulose. This suggests the possibility of a 1:1 relationship between the SCD and the arabinose in oxidized cellulose.

With the foregoing in mind, the ratio of SCD to arabinose in sample 2 of oxidation II was evaluated and found to be about 1.7:1. This result has two possible interpretations: (1) all of the SCD in the oxidized cellulose was not entirely associated with the carbon 1 position and the carbonic acid ester linkage, or (2) the arabinose formed by the oxidative cleavage of carbons 1 and 2 of the anhydroglucose unit was subsequently oxidatively degraded, while the carbonic acid ester groupings, formed by the oxidation, either remained intact or was hydrolyzed at a slower rate than the arabinose was degraded. Therefore, due to the uncertainty of the meaning of the SCD-arabinose ratio obtained for this oxidized cellulose and to the lack of a truly satisfactory method for accurately quantitating small quantities of arabinose in oxidized cellulose, less than 0.2% by weight, this approach to the study of the carbonic acid ester hypothesis showed little promise and was abandoned.

RADIOCHEMICAL STUDIES

The isolation of the SCD from acid chlorine-oxidized isotopically-labeled cellulose and the characterization of this carbon dioxide as to its origin, carbon 1, 2, etc. of the anhydroglucose molecule, constitute a direct test of the validity of Kaverzneva's carbonic acid ester linkage (see Fig. 1). If the SCD were shown to consist of carbon dioxide derived exclusively from carbon 1, the hypothesis would be greatly strengthened. However, if carbon 1 were shown to represent little or none of the SCD, the probability of the existence of the postulated stable ester linkage would be reduced; such a negative

Table III. Summary of Results—Saponifiable Carbon Dioxide Found in Acid Chlorine-Oxidized Labeled Bacterial Cellulose

Item number	*Sample identification*	*Buffer system (pH 4.3 ± 0.3)*	*Oxidant consumption equiv. HOCl/mole anhydroglucose*	*Carbon dioxide liberated during oxidation, %[a]*	*Specific activity of CO_2 liberated during oxidation, c.p.m./mg. C*	*Yield of oxidized cellulose, %*	*SCD mole CO_2/100 moles anhydroglucose*	*Specific activity of SCD, c.p.m./mg. C*	*Maximum of saponifiable carbon dioxide (SCD) originating at the C(1)-position,[b] %*
1	Oxidation VI Batch 3	Acetate	0.29	0.61	1039	96.2	0.069 (one-hour saponification)	1915	13.7
							0.102 (12-hour saponification)	2015	14.5
2	Oxidation VII Batch 3	Acetate	0.82	2.09	1352	89.7	0.154, 0.149 } 0.15	2420, 2345 } 2382	17.1
3	Oxidation XII Batch 3 (free of included solvents)	Phosphate	0.91	1.99[c]	2841[c]	89.8	0.143, 0.154 } 0.15	2041, 2063 } 2052	14.7
4	Oxidation VIII Batch 3	Acetate	1.37	4.65	1348	86.9	0.173, 0.168 } 0.17	2262, 2305 } 2283	16.4
5	Oxidation V Batch 1	Acetate	2.65	..	..	84.1	0.261, 0.237 } 0.25	2736, 2825 } 2774	20.8
6	Oxidation IX Batch 4	Acetate	1.38	5.10	728	86.1	0.173, 0.181 } 0.18	1582, 1626 } 1604	16.0[d]

[a] Based on original cellulose.
[b] Limitation valid provided assumptions made with respect to isotope effect and label distribution hold.
[c] These represent only valid values in these columns; other values are in error due to included solvent.
[d] For this sample, the maximum SCD originating at position 6 is 17%.

finding would also serve to open the way for consideration of the true source of the SCD in acid chlorine-oxidized cellulose.

C^{14}-labeled cellulose was biosynthesized by *Acetobacter xylinum* from both D-glucose-1-C^{14} and D-glucose-6-C^{14}. These labeled bacterial celluloses were prepared in yields of 35 to 40% on the basis of the glucose supplied and 16 to 21% on the basis of the carbon-14 supplied (see Table II). The labeled celluloses were not entirely specific despite the use of specifically labeled glucose; this was due to the entry of part of the labeled glucose into the metabolic process of the organism (*35*). The label in the cellulose-1-C^{14} was 80% specific at carbon 1 and 2% specific at carbon 6; these values compared favorably with those reported by Greathouse (*17*). For the cellulose-6-C^{14}, the label was 24% specific at carbon 1 and 59% specific at carbon 6; whereas Greathouse reported an 82% specificity in position 6 of a similarly prepared labeled cellulose. This low result was probably due to one or both of the following:

1. The nutrient medium for this preparation was contaminated with unknown species of bacteria, as evidenced by abnormal turbidity and color of the medium.

2. Greathouse's method of preparation was not adhered to exactly; the alcohol specified for the nutrient medium was not used.

Although the specificity of the labeled celluloses was less than 100% it is felt that both preparations were adequate for the support of the conclusions drawn from the studies described below.

Cellulose-1-C^{14} was oxidized to five different levels of oxidation (oxidations V–VIII, and XII); the oxidant consumption for these oxidations ranged from 0.29 to 2.65 equiv. hypochlorous acid per mole of anhydroglucose. The C(1)-labeled celluloses used had a specific activity in position 1 ranging from about 13,300 to 13,900 c.p.m. per mg. (see Table II). The average specific activity of the SCD from the oxidized C(1)-labeled celluloses ranged from 2015 to 2774 c.p.m. per mg. C (see Table III). On the basis of the specific activity of the carbon in position 1 of the cellulose-1-C^{14} that was oxidized, the maximum percentage of the SCD from the samples that could have originated at position 1 was 14.5 to 20.8%. In reality, the contribution of position 1 could never have reached the maximums listed above, for all the positions of the anhydroglucose molecule had some label. The quantity and specific activity of the SCD from a given oxidized cellulose were reproducible.

The effect of saponification time was checked on samples from oxidation VI. The quantity of SCD liberated by 1-hr. treatment was less than that liberated by a 12-hr. treatment, 0.07 as opposed to 0.10 mole CO_2 per 100 moles of anhydroglucose. The specific activities of the SCD liberated by these different saponifications were comparable, 1915 and 2015 c.p.m. per mg. C, respectively. Therefore, it seems likely that the source of the SCD was in both cases about the same.

A sample of cellulose-6-C^{14} was oxidized (oxidation IX) to a level of oxidation comparable to that of oxidation VIII; the specific activity of the SCD from this oxidized cellulose-6-C^{14} was 1604 c.p.m. per mg. C. By assuming that the SCD from the two oxidized celluloses, oxidations VIII and IX, had the same origin and that any carbon dioxide derived from positions other than 1 and 6 was of zero activity, the maximum contributions of positions 1 and 6 were calculated as 16 and 17%, respectively. Of course, one or both of these values is high, for the second assumption made above is known to be erroneous. Only one oxidation was made using cellulose-6-C^{14}, so the analysis of the part played by position 6 may be considered as preliminary.

POSTULATED SOURCES OF SAPONIFIABLE CARBON DIOXIDE

It follows from above that there may be some carbonic acid ester linkages at position 1 in acid chlorine-oxidized cellulose, but the origin of most of the SCD is not known. There are two broad explanations for this SCD; these are:

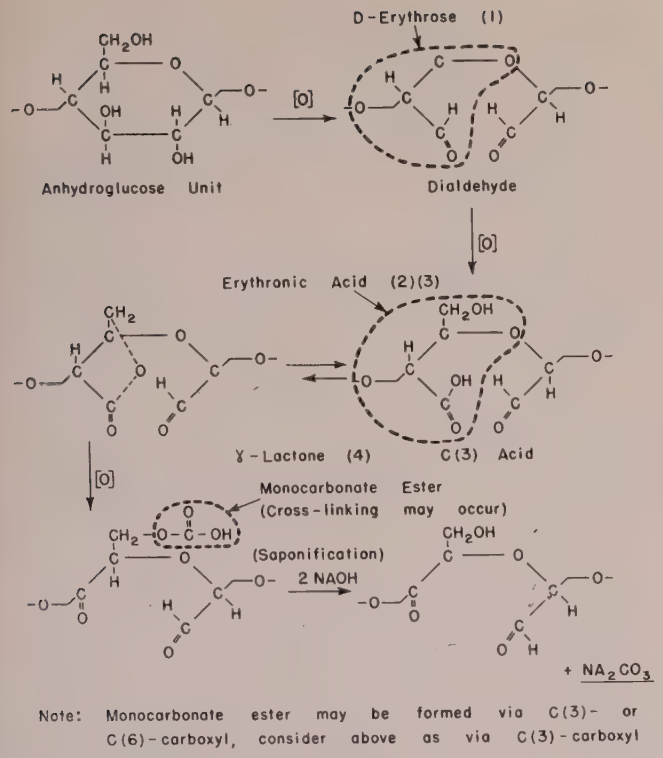

Fig. 5. Monocarbonate ester hypothesis (γ-lactone intermediate)

1. Carbon dioxide liberated during the oxidation of cellulose may be physically absorbed or included in the cellulose, with these physical bonds or obstructions being labile to dilute alkali.

2. Some structure or structures, not involving position 1, that liberate carbon dioxide when subjected to the saponification treatment are present in acid chlorine-oxidized cellulose.

(Both of these may occur simultaneously.)

The specific activity of the carbon dioxide liberated during the oxidation was not directly related to the specific activity of the SCD; this shows that the SCD was not due to only adsorption or inclusion of part of this carbon dioxide in the cellulose. This possibility was disproved by the following experiment.

Radioactive carbon dioxide with a specific activity of about 12,000 c.p.m. per mg. C was continuously cycled through the oxidation mixture throughout the oxidation of an unlabeled bacterial cellulose (oxidation XI). When the bulk of the oxidant was consumed, cycling of the radioactive carbon dioxide was discontinued, and the oxidized cellulose was washed and solvent dried. The specific activity of the SCD in the oxidized cellulose was then determined; it was found to be approximately zero.

Inasmuch as the carbonic acid ester (position 1) can make only a minor contribution (21%) to the SCD in acid chlorine-oxidized cellulose, it is necessary for other structures, not associated with position 1, to make up the major part of the SCD, i.e., if chemical structures are solely responsible for the SCD. Hypotheses for the formation of monocarbonate and formate esters are presented above. These structures for the most part are speculative and are by no means felt to be the only ones possible.

The postulated monocarbonate ester grouping may be formed from carbon 3 or 6; the formation involves a γ-lactone intermediate or the C(3)- or C(6)-acid. The mechanism depicted in Fig. 5 is for the formation of the monocarbonate ester from carbon 3; supporting evidence for this follows: (1) The presence of erythrose in acid chlorine-oxidized cellulose has been chromatographically verified in this research. (2) Jayme and Maris (*36*) have suggested that the aldehyde group at position 3 reacts much faster than does that at position 2. (3) Erythronic acid has not been identified in acid chlorine-oxidized cellulose, but it has been identified by Whistler and Schweiger (*37*) in acid chlorine-oxidized amylopectin. (4) Kaverzneva, *et al.*, (*5*), showed the presence of lactones in acid chlorine-oxidized cellulose. (5) γ-Lactones are known to be very stable in acidic media; however, it is not known whether an oxidative carbon-carbon cleavage could take place leaving the lactone group intact. One theory that may support such a cleavage is that pertaining to the oxidative cleavage of a carbon-carbon bond with lead tetraacetate leaving a lactol linkage intact; this theory was advanced by Perlin and Brice (*38*) for the lead-tetraacetate oxidation of sugars. (6) The stability and reactivity (ability to cross link) of a monocarbonate ester of the type shown are not known; however, monocarbonate esters are normally rather unstable (*39*). (The above supporting evidence, where applicable, may be construed to cover monocarbonate formation via the C(6)-carbon.)

The formation of formate esters via lactol intermediates (carbons 2, 3, and/or 6) may be hypothesized. These formate esters would be saponifiable in the presence of dilute alkali, thus forming sodium formate which on acidification would be converted to formic acid. The formic acid may be readily decomposed to carbon dioxide in the presence of the reducible groupings in the oxidized cellulose. Metal ions present in the acidic cellulose suspension could catalyze such a reaction.

(The specific activity of the SCD found in the acid chlorine-oxidized celluloses may be explained on the basis of various combinations of the structures hypothesized above.)

Examination of Carbon Dioxide Evolved During the Oxidation of Labeled Bacterial Cellulose

The specific activity of the carbon dioxide evolved during oxidations VI through IX (ranging from 13,300 to 13,900 c.p.m. per mg. C for the oxidations of cellulose-1-C^{14}) was in error due to the presence of solvent included during the solvent drying of the purified cellulose. This nonradioactive organic material (about 4%), principally diethyl ether with perhaps some acetone and ethyl alcohol, was shown to be present by the lower specific activity of the cellulose (1841 c.p.m. per mg. C) as compared with that of potassium gluconate prepared from the cellulose (1920 c.p.m. per mg. C). The above-mentioned solvents were found to liberate some carbon dioxide due to the oxidative action of aqueous chlorine at pH 4.5. The acetate buffer system used in these oxidations was also oxidizable to carbon dioxide by aqueous chlorine at pH 4.5.

The solvent was removed from a sample of solvent-dried, purified cellulose-1-C^{14} (batch 3) by boiling it in water for 3 hr. The cellulose was filtered from the water and was washed with boiling water. This

Table IV. Uronic Acid Determinations

Sample identification	*"Uronic acid" carbon dioxide evolved, %*	*"Uronic anhydride," %*	*Specific activity of "uronic acid" carbon dioxide, c.p.m./mg. C*
(1) Unoxidized bacterial cellulose: Cellulose-1-C^{14} (Batch 3)	0.10	0.40	4968
(2) Unoxidized bacterial cellulose: Cellulose-6-C^{14} (Batch 4)	0.13	0.52	1109
(3) Unoxidized purified cotton[a]	0.07	0.27	..
(4) Oxidized bacterial cellulose: Before saponification	0.20	0.80	3698
Cellulose-1-C^{14} (Oxidation VI, 0.29 equiv. HOCl/mole anhydroglucose) After saponification	0.20	0.80	3609
(5) Oxidized bacterial cellulose: Before saponification	0.41	1.64	3199
Cellulose-1-C^{14} (Oxidation VII, 0.82 equiv. HOCl/mole anhydroglucose) After saponification	0.38	1.52	2925
(6) Oxidized bacterial cellulose: Cellulose-1-C^{14}(Oxidation XII, 0.91 equiv. HOCl/mole anhydroglucose)	0.43	1.72	2825
(7) Oxidized bacterial cellulose: Cellulose-1-C^{14} (Oxidation VIII, 1.4 equiv. HOCl/mole anhydroglucose)	0.62	2.48	1698
(8) Oxidized bacterial cellulose: Cellulose-6-C^{14} (Oxidation IX, 1.4 equiv. HOCl/mole anhydroglucose)	0.57	2.28	1790
(9) Oxidized cotton[a]: (0.26 equiv. HOCl/mole anhydroglucose)	0.29	1.14	..
(10) Oxidized cotton[a] (0.89 equiv. HOCl/mole anhydroglucose)	0.52	2.08	..

[a] Data reported by Kaverznva, *et al.* (*5*).

NOTE: (1) All the values reported above that were obtained in this research are based on duplicate determinations with a deviation from the average ranging from ± 1 to 5%. (2) "Uronic acid" carbon dioxide values are low by as much as 7% due to error in the modified Browning method (*20*) that was used.

cellulose was oxidized in a phosphate buffer system (oxidation XII). Carbon dioxide with a specific activity of 2841 c.p.m. per mg. C was evolved from the oxidation mixture. This result showed the effect that the included solvent and acetate buffer system had had in the previous oxidations. The specific activity of the carbon dioxide evolved during this oxidation approached that of the cellulose-1-C^{14} (2914 c.p.m. per mg. C) that was oxidized.

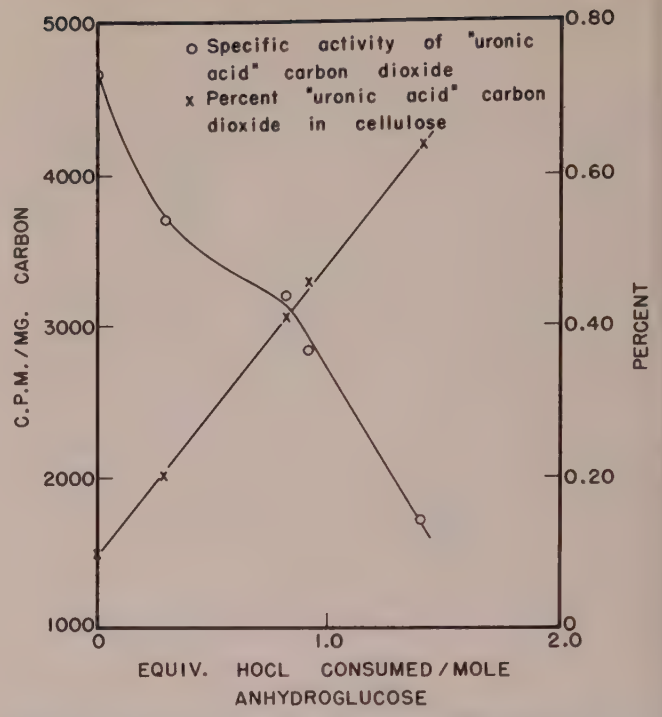

Fig. 6. "Uronic acid" carbon dioxide from cellulose-1-C^{14} (batch 3) at various oxidation levels

It is believed that the bulk of the carbon dioxide evolved during this apparently random oxidation was derived from subsequent oxidation of solubilized fragments of the cellulose.

THE QUANTITY AND RADIOACTIVITY OF "URONIC ACID" CARBON DIOXIDE IN LABELED OXIDIZED AND UNOXIDIZED BACTERIAL CELLULOSES

The specific activity values of the "uronic acid" carbon dioxide from cellulose-1-C^{14} at various levels of oxidation ranged from about 5000 down to 1700 c.p.m. per mg. C. Therefore, the "uronic acid" carbon dioxide did not have the C(6)-position as its only point of origin, because the specific activity in position 6 of the cellulose-1-C^{14} used was only 362 c.p.m. per mg. C. The specific activity decreased as the level of oxidation and the per cent of "uronic acid" carbon dioxide in the cellulose increased (see Fig. 6). This shows a change in the fraction of the carbon dioxide coming from the various positions in the cellulose as a function of oxidation level.

At least 30% of the "uronic acid" carbon dioxide from the unoxidized cellulose-1-C^{14} (item 1, Table IV) was derived from position 1. The foregoing suggests some decomposition of the glucose hydrolyzed from the cellulose, by the 12% HCl, with the liberation of the C(1)-position as carbon dioxide. On analysis of items 7 and 8 in Table IV, it is apparent that no more than about 22% of the "uronic acid" carbon dioxide from these oxidized celluloses came from the C(6)-position and no more than 12% came from the C(1)-position. Thus, positions 2 and 3 of the cellulose undoubtedly contribute substantially to the "uronic acid" carbon dioxide from this type of oxidized cellulose at this level of oxidation. The carbon dioxide originating at positions 2 and 3 may be derived from erythronic

Table V. Cellulose Crystallinities

Sample identification	Measure of crystallinity: Degree of crystallinity (DCh)[a]	Measure of crystallinity: Crystalline index (CrI)[b]
(1) Purified unoxidized cotton	0.84	0.78
(2) Oxidized cotton sample, oxidation II	0.85	0.81
(3) Unoxidized cellulose-1-C^{14}, batch 3	0.86	0.79
(4) Oxidized cellulose-1-C^{14}, oxidation VIII	0.87	0.82
(5) Unoxidized cellulose-6-C^{14}, batch 4	0.87	0.81
(6) Oxidized cellulose-6-C^{14}, oxidation IX	0.87	0.82

[a] Degree of crystallinity according to Hermans and Weidinger as interpreted by Ant-Wuorinen (*43*).
[b] Ant-Wuorinen's crystalline index (*43*).
Note: All samples were at density of 1.40 ± 0.02 g./cc.

or glyoxylic acids or from some type of ketonic or aldehydic structure that is unstable under the drastic conditions of the uronic acid determination.

COMPARISON OF UNOXIDIZED AND OXIDIZED COTTON AND BACTERIAL CELLULOSES

As has been noted earlier, bacterial cellulose responds to acid chlorine oxidation in about the same manner as does cotton. However, before bacterial cellulose was utilized in this research, a number of physical and chemical comparisons were made between it and cotton cellulose to ascertain the soundness of extending to both celluloses interpretations made on the basis of results obtained for only one type. These studies were patterned after those made by Hibbert and Barsha (*40*), Greathouse and Shirk (*41*), and Schramm and Hestrin (*42*); (the findings of these men support the conclusion that the chemical properties of bacterial and cotton celluloses are essentially identical).

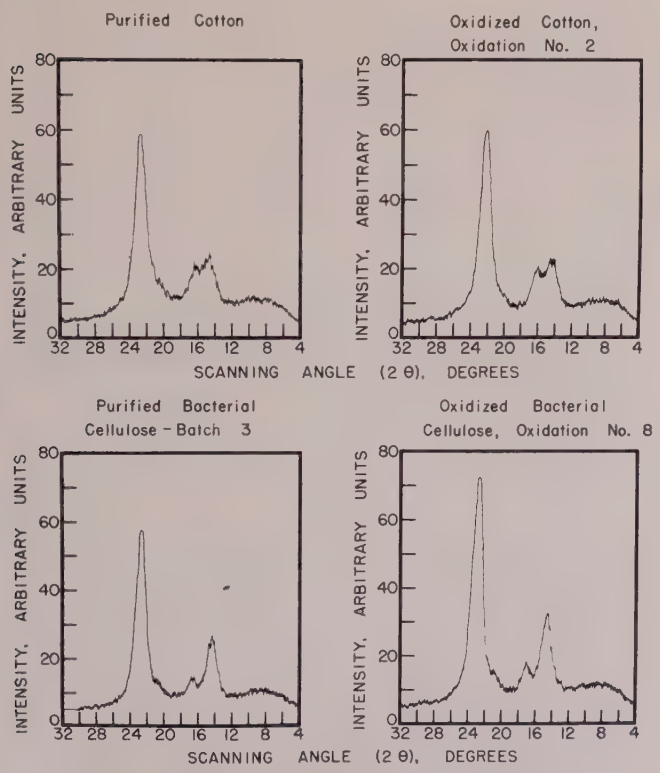
Purified Cotton
Oxidized Cotton, Oxidation No. 2
Purified Bacterial Cellulose - Batch 3
Oxidized Bacterial Cellulose, Oxidation No. 8
INTENSITY, ARBITRARY UNITS
SCANNING ANGLE (2 θ), DEGREES

Fig. 7. X-ray diffraction patterns of unoxidized and oxidized cotton and bacterial celluloses

Physical Properties

X-ray analyses were made on both oxidized and unoxidized cotton and bacterial celluloses (see Fig. 7). In each case, the major reinforcement angles were found at 2θ values of 14.5°, 16.8°, and 22.5°. Crystallinity, according to Ant-Wuorinen (*43*) and Hermans and Weidinger as interpreted by Ant-Wuorinen (*43*), was calculated from the X-ray data for each sample studied. The degree of crystallinity (DCr) according to Hermans and Weidinger is interpreted as an index of the per cent of crystallinity of cellulose. The crystalline index (CrI) of Ant-Wuorinen is interpreted as an index of the degree of order of the crystalline region of cellulose (see Table V).

The cotton and bacterial celluloses were found to have about the same per cent crystallinity and crystalline order; the bacterial cellulose was slightly higher for both. A minor increase in the per cent crystallinity and crystalline order was noted for each sample after oxidation; this was probably due to a preferential removal of the amorphous and less-ordered crystalline regions of the cellulose by the oxidation.

Infrared Spectra. Infrared spectra were obtained for oxidized and unoxidized cotton and bacterial celluloses. On comparison, these spectra were found to be greatly similar, with the exception that at 13.3 mu (750 $cm.^{-1}$), the bacterial celluloses had an absorption band not observed in the spectrum for cotton. This band may result from absorption caused by structural configurations that are found in many amino acids (*44, 45*) (see Fig. 8). The infrared spectra of oxidized cotton and bacterial celluloses had an absorption band at 5.75 mu (1740 $cm.^{-1}$). The new band found in the oxidized celluloses may be attributed to either aldehydic, ketonic, lactone, or ester carbonyl stretch vibrations (*44*).

Chemical Properties

Glucose Content. The hydrolyzates of purified,

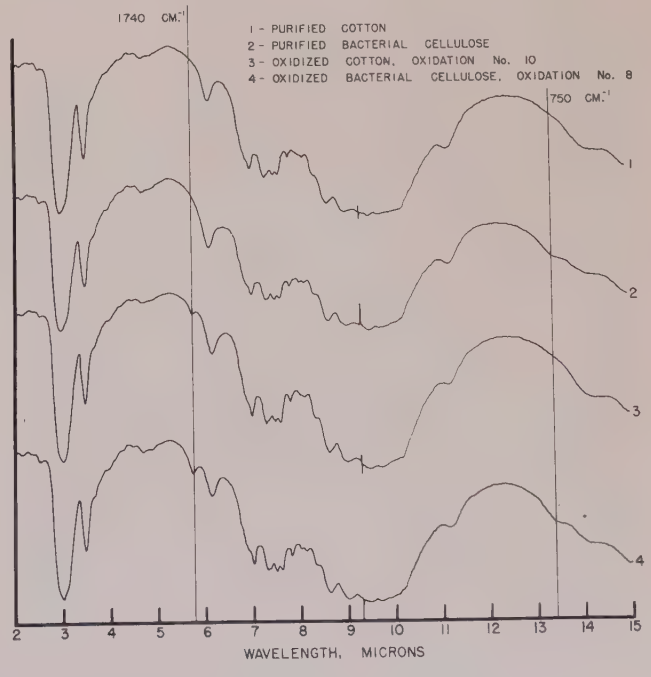
1740 CM.⁻¹
750 CM.⁻¹
1 - PURIFIED COTTON
2 - PURIFIED BACTERIAL CELLULOSE
3 - OXIDIZED COTTON, OXIDATION No. 10
4 - OXIDIZED BACTERIAL CELLULOSE, OXIDATION No. 8
WAVELENGTH, MICRONS

Fig. 8. Infrared absorption spectra of unoxidized and oxidized cotton and bacterial celluloses

Table VI. Acid Chlorine-Oxidized Bacterial Cellulose Alkali Sensitivity Study

Sample identification	Oxidant consumption, equiv. HOCl /mole anhydroglucose	D.P. (cellulose nitrate in acetone)	D.P. (cellulose in CED)	ΔD.P.	Alkali-labile linkages/1000 anhydroglucose units	SCD, mole CO_2/1000 moles anhydroglucose
(1) Unoxidized cellulose-1-C^{14} (Batch 3)	..	3370	3370	..	..	..
(2) Unoxidized cellulose-6-C^{14} (Batch 4)	..	2750	2750	..	..	..
(3) Oxidized cellulose-1-C^{14} (Oxidation VI)	0.29	1090	622	468	0.85	1.0
(4) Oxidized cellulose-1-C^{14} (Oxidation VIII)	0.82	813	422	391	0.95	1.5

unoxidized cotton and bacterial celluloses contained largely glucose with some oligosaccharides; the bacterial cellulose hydrolyzate also contained a trace of an unidentified amino acid. The glucose in the bacterial cellulose hydrolyzate represented 87% by weight of the original purified cellulose. In the light of the quantity of oligosaccharides remaining in the hydrolyzate, this value compares favorably with values previously reported for purified cotton, e.g., the 93% reported by Schramm and Hestrin (*42*). From this result, it is apparent that bacterial cellulose is composed primarily of glucose linked by β 1–4 linkages; the β-linkage was checked by hydrolysis with emulsin.

Nitrogen Content. The nitrogen content of oxidized and unoxidized cotton and bacterial celluloses was determined according to the colorimetric method of Niss (*46*). The per cent nitrogen in the celluloses was low: bacterial cellulose, 0.04 to 0.20%; cotton, 0.02 to 0.03%; nitrogen content (%) dropped with degree of oxidation. Proteinaceous material probably accounts for the bulk of the nitrogen in both types of cellulose.

Cellulose Nitration. Bacterial celluloses gave lower yields of cellulose nitrate than are normally obtained with cotton—95% as opposed to 98 to 99%. In spite of the fact that the bacterial cellulose nitrate yields were 5% below theoretical, the results obtained, in view of the high degree of nitration (13.9%), are quite valuable, for they show rather conclusively that the bacterial cellulose is at least 95% cellulosic.

Alkali Sensitivity. Alkali sensitivity of acid chlorine-oxidized cotton cellulose has long been recognized (*11*). Thus, as another means of noting the similarity of cotton and bacterial celluloses, a study was made to determine the alkali sensitivity of acid chlorine-oxidized bacterial cellulose; it showed that oxidized bacterial cellulose also contains alkali-labile linkage (see Table VI).

The data presented in Table VI show that the structures in acid chlorine-oxidized cellulose that liberate SCD are not exclusively associated with alkali-sensitive linkages, i.e., groupings capable of cellulose chain cleavage, for example, the hypothesized carbonic acid ester linkage. On the basis of these and previously discussed data, the carbonic acid ester groupings could be responsible for only a minor part of the alkali-labile structures causing chain cleavage in acid chlorine-oxidized cellulose.

Conclusion

From the foregoing comparison of physical and chemical properties of unoxidized and acid chlorine-oxidized cotton and bacterial celluloses, it was evident that these two celluloses could be used interchangeably, if qualitative data were required. Therefore, the usefulness of labeled bacterial cellulose as a tool for cellulose oxidation research seemed valid so long as the following assumptions held: (a) the *isotope effect* is negligible, (b) *intra-polymer distribution* of labeled anhydroglucose units is random, and (c) the portions of the polymer that are accessible to oxidation are *randomly oxidized.*

CONCLUSIONS

1. The SCD in acid chlorine-oxidized cellulose is not derived from carbon in a single position in the anhydroglucose molecule. Position 1 can be the source of not more and probably less than 21% of the SCD. Therefore, the probability of the existence of the hypothesized carbonic acid ester linkage is greatly reduced.

2. Some chemical structure or structures, not involving position 1, are responsible for the SCD that cannot be accounted for by the carbonic acid ester linkage.

3. The carbonic acid ester linkage does not play a significant role in the alkali sensitivity of acid chlorine-oxidized cellulose.

4. The application of the uronic acid determination to unoxidized or to acid chlorine-oxidized cellulose is meaningless when interpreted in terms of C(6)-carboxyl, for much of the "uronic acid" carbon dioxide does not originate at the C(6)-position. The fraction of the "uronic acid" carbon dioxide coming from the various positions in a cellulose changes as a function of the oxidation level of the cellulose. Hence, small changes in the uronic acid content of acid chlorine-oxidized celluloses defy precise interpretation.

5. The SCD in acid chlorine-oxidized cellulose is not responsible for a significant amount of the pseudouronic acid carbon dioxide-yielding groups present in these celluloses.

6. The net interpretation of all the present results is that the acid chlorine-oxidation of cellulose is nonspecific, i.e., the structure of acid chlorine-oxidized cellulose cannot be precisely formulated on the basis of a single primary point of oxidation.

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A Method of Determining the Substance over Small Areas of Paper

E. S. BRAZINGTON and B. RADVAN

Photographs of several samples of paper, obtained by means of alpha rays, emitted from a Polonium 210 source, are presented. Since the range of these rays is constant at approximately 24 g. per sq. in., each exposed spot on the photographs corresponds to an area of substance less than that value. By interposing thin films of aluminium between the source and the paper, a further series of photographs have been obtained, in which the exposed spots correspond to arrears of increasingly lower substance. The main limitations of the method lie in its stepwise nature, and the inevitable lack of collimation. The apparatus is cheap and simple, but safety precautions must be observed in its use.

The direct methods of determining the substance, by weighing, become troublesome for areas of less than some inches across. The least size of an area, over which substance can be measured by means of the several types of beta ray gages, now available, is also of the same order. Indirect determinations over areas smaller than this are usually referred to as measurements of "formation"—they record variations in the transmission of light as an index of the corresponding variations in substance (*1*).

A new direct method of measuring substance over very small areas—some $^1/_2$ mm. across—is provided by the use of alpha ray photography. The method was invented at the Isotope Division, AERE, Harwell, U.K., in response to a request from the Wiggins Teape Group Research Organisation, and was then further developed at these laboratories.

The principle of the method relies on the uniform distribution of energy of alpha particles emitted from a radioactive source, which results in all of them having practically the same range (*2*) in a given material. This may be contrasted with the transmission of light through transparent materials, where a constant proportion is absorbed in each layer, so that no range may be discerned, or with the behavior of beta-particles, the range of which is very ill defined (*2*). Thus, a photograph of light transmitted through a sample of paper will present a cloudy appearance, the intensity of exposure varying continuously with the transparency of different areas.

With alpha rays, such a photograph is much simpler;

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those parts of the sample over which the substance is less than the range, will be exposed to nearly uniform intensity, and those where the substance exceeds the range will not be exposed at all. Now, the effective range may be reduced by interposing a uniform obstruction, such as a thin foil of aluminium, between the

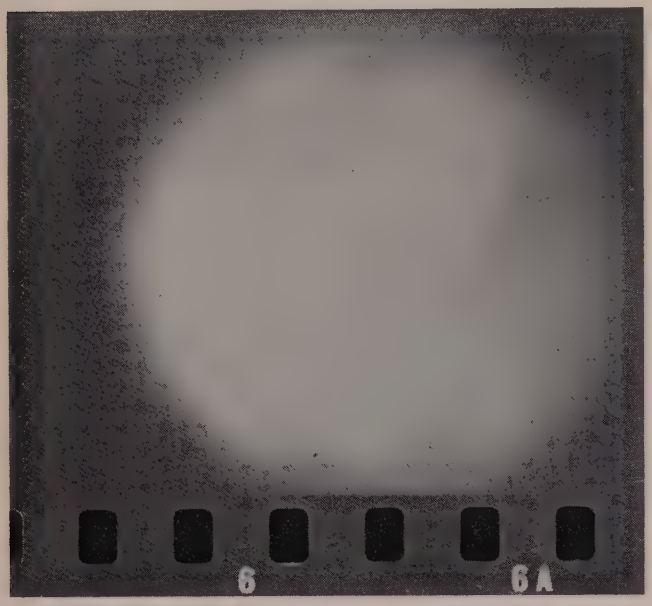

Fig. 1

source and the sample, when a larger proportion of the area of the sample, of substance exceeding the new reduced range, will now remain unexposed. Repeating the procedure several times produces a series of "sections" through the sample, each at a different level of substance.

The apparatus used in the work described here consists, in essence, of three parts; a radioactive source,

Fig. 2

a 35-mm. contact print camera, and a turntable with aluminium foil "windows" inset in it. The source consists of a layer of a Polonium 210 salt, deposited over an area of $1^1/_2$ in. diam., covered with a thin sheet of mica and mounted in a brass holder. The holder is screwed into a stand, the sample is stretched tightly at

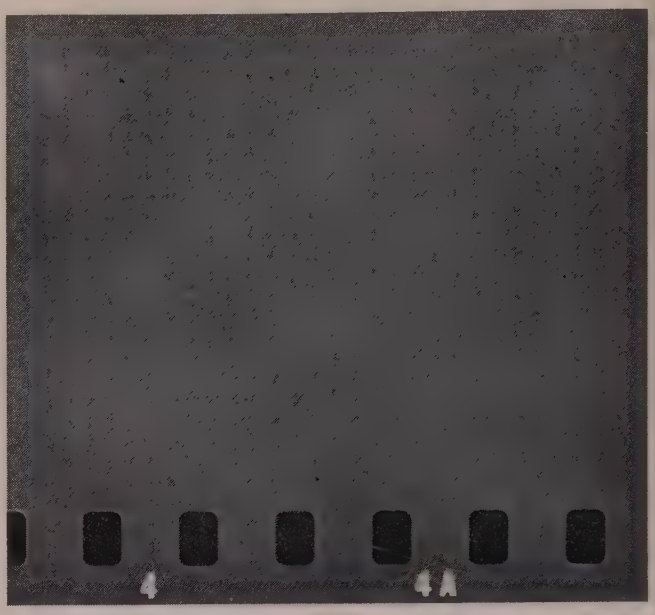

Fig. 3

a distance of 0.4 cm. above the source, and the camera is then placed with the film in taut contact with the sample. The turntable is now rotated so as to interpose the window with the required thickness of aluminium foil between the source and the sample, and the film is exposed for a set length of time. The film is then

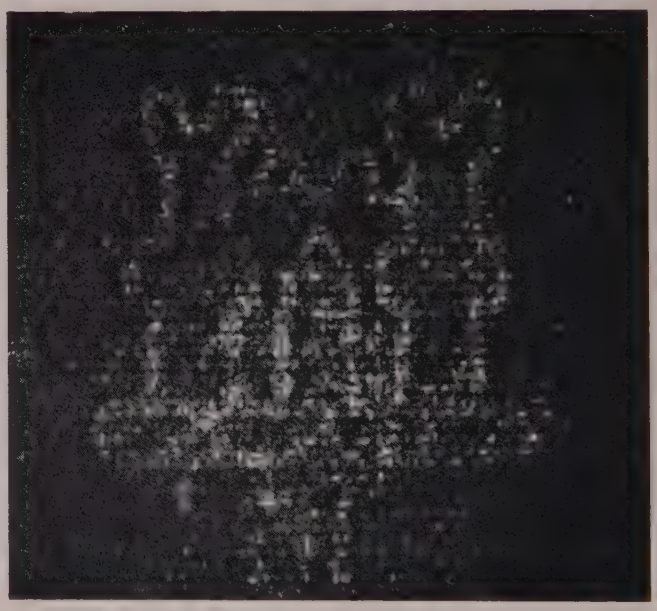

Fig. 4

wound forward, the turntable rotated to present a different foil, etc.

Throughout the procedure, safety precautions have to be observed. Thus, the source must be handled with some care, in order to avoid rupturing the thin covering of mica, which would result in a spillage of

the highly dangerous radioactive dust. The alpha rays emitted from the source are not dangerous in practice taken that they do not impinge upon any vital part of

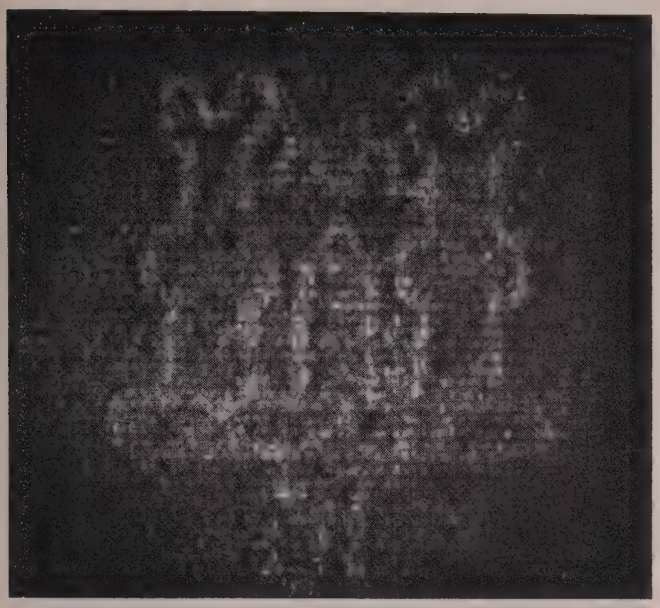

Fig. 5

the body. Otherwise the apparatus is both cheap and simple.

Two sources have been used, both supplied by the Radiochemical Centre, Amersham, U.K., one of a strength of 2 millicuries, the other 10 millicuries (half life approx. 6 months). Exposure times varied from 30 sec. to 15 min., depending on the strength of

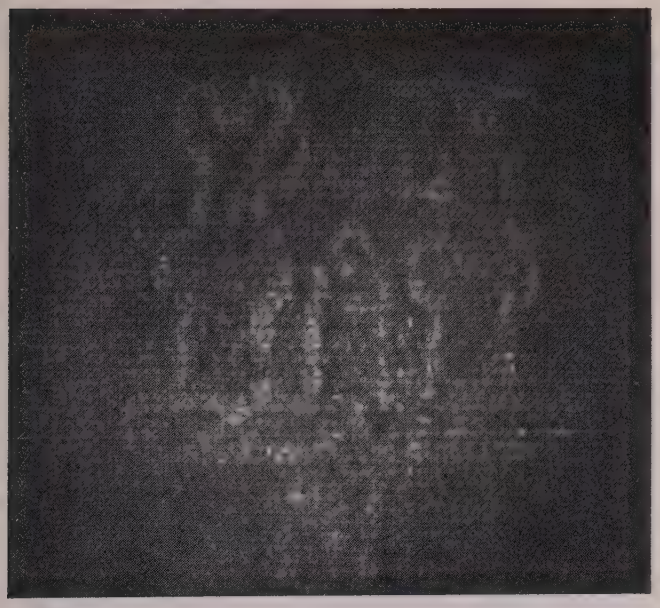

Fig. 6

the source, and weight of the samples. Moderately fast film (Ilford F.P.3) was used in most experiments; a faster one (Ilford H.P.S.), when the times of exposure became inconveniently long.

Some of the results are illustrated by the photographs, all of which are positives. Figures 1, 2, and 3 are 10 min. exposures (enlarged 2.4 ×), with no sample between the source and the film, but only aluminium foils of weights 35.5, 40.0, and 43.0 g. per sq.m., respectively. Now 11 g. per sq. m. of aluminium has the same stopping power as 7 g. per sq. m. of alpha-cellulose. It will be seen that a "cut off" occurred

Fig. 7

between foils of weights equivalent to 22.6 and 25.5 g. per sq. m. of cellulose. Thus, in every succeeding photograph, an exposed spot may be taken to represent an area where the combined "substance" of paper and foil was less than 24 g. per sq.m.

The other photographs are listed in Table I (enlarged 3.2×) (Figs. 4–11).

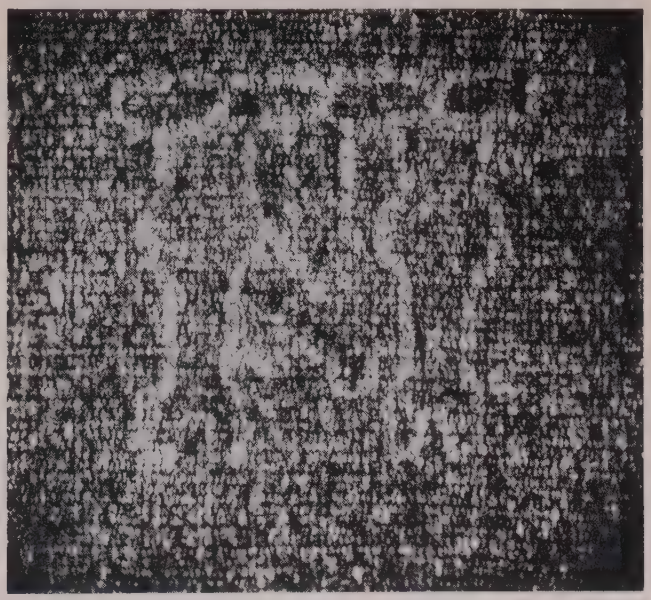

Fig. 8

An obvious limitation of the method results from the stepwise changes in the thickness of aluminium foils; thus, the substance "sections" are separated by some

2 to 3 g. per sq.m., at least. Another source of inaccuracies is the lack of uniformity of the commercially available aluminium foil. However, Figs. 1 to 3

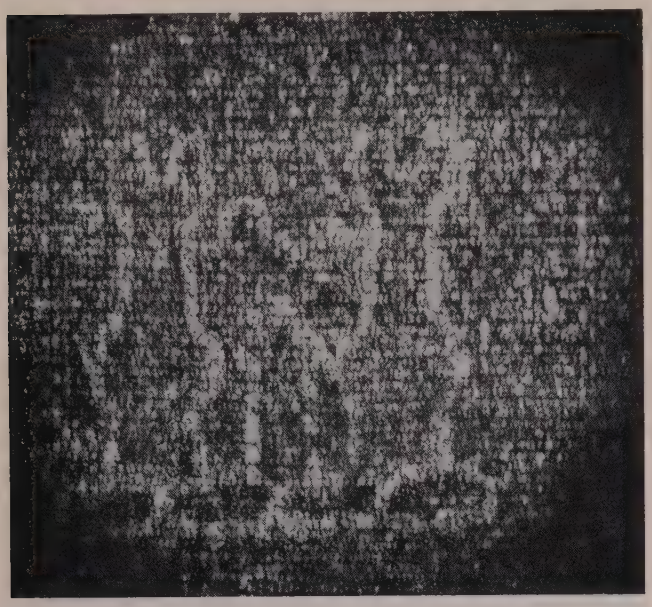

Fig. 9

show that these variations are of a smaller order of magnitude than the size of the steps.

Another inaccuracy results from the uneven distribution of radioactivity over the area of the source. This is particularly troublesome with sources of higher strength, and results in uneven intensity of exposure over different parts of the print. It should be empha-

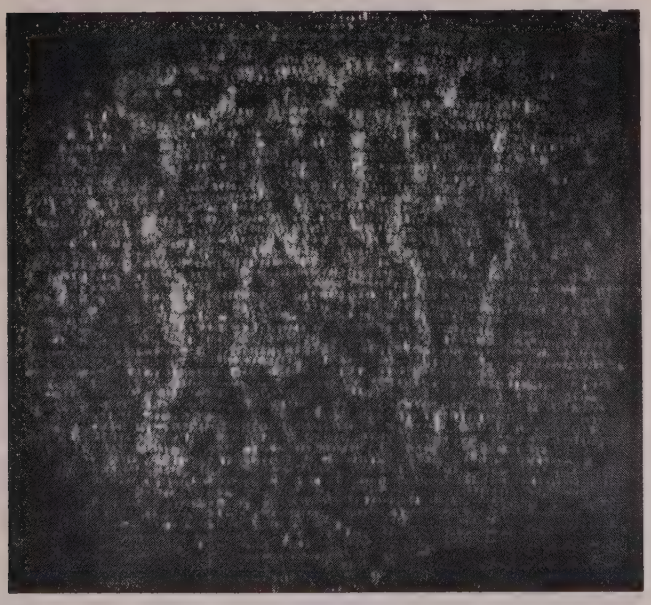

Fig: 10

sized, however, that the range of alpha particles is not affected by the strength of the source. Also, very strong sources emit other forms of radiation, of sufficient intensity to produce a faint exposure, even when alpha range is exceeded. This difficulty can be overcome by a judicious choice of the times of exposure. A more serious error arises from the lack of collimation, and may only be overcome through bringing the source as close to the sample as is safely permissible. It will also be noticed that, the maximum range being of the order of 25 g. per sq. m., only comparatively light-

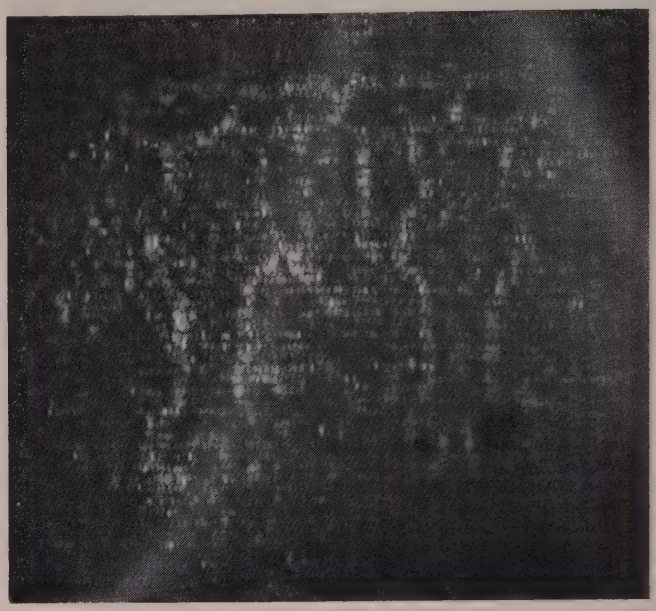

Fig: 11

weight papers may be usefully examined by this method.

Table I

Figure no.	*Subject*	*Light spots represent areas of weight less than, g./sq. m.*	*Exposure, min.*
4	45 g./sq. m.—Bank	24	2
5	45 g./sq. m.—Bank	20	
6	45 g./sq. m.—Bank	17	
7	45 g./sq. m.—Bank	14	
8	28 g./sq. m.—Airmail	24	2
9	28 g./sq. m.—Airmail	21	
10	28 g./sq. m.—Airmail	17	
11	28 g./sq. m.—Airmail	15	

Finally, it may be mentioned that the same apparatus, slightly modified, may be used to obtain a numerical distribution of substance and area over the sample. The principle of this method is explained in (*3*); it entails counting of the transmitted particles, rather than recording them on a photographic film. Work on this approach is now proceeding.

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2. Whitehouse, Putman, "Radioactive Isotopes," Clarendon Press, 1953. See pp. 64–73.
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This method was developed at AERE, Harwell, and in these laboratories. The authors wish to express their thanks to the Board of Directors of Wiggins Teape & Co. Ltd., in particular, H. F. Rance, for the permission to publish this paper

Effect of Time and Temperature on the Direct Nitration of *Eucalyptus Rostrata*

MENACHEM LEWIN and JOSEPH A. EPSTEIN

Samples of wood of *Eucalyptus rostrata* (river red gum) were nitrated for various intervals of time from 1/2 to over 1000 hr. at 3°C. and the products isolated and examined. The following fractions were obtained: A, the crude nitrate which was further divided into A1, the acetone soluble fraction precipitated by water, A2, the acetone insoluble fraction, and A3, the acetone and water-soluble fraction. The results are compared to those obtained previously for nitration at 20°C., and are further interpreted in terms of the alpha-cellulose-hemicellulose bond which was postulated for this wood. The temperature coefficient and energy of activation of the decomposition of this bond is calculated. The degree of polymerization of the various A1 fractions is determined and its mode of variation explained in terms of the above postulate. The value of the direct nitration of this wood as an analytical tool for the determination of alpha-cellulose, degree of polymerization of the cellulose and its distribution, is discussed.

THE method of direct nitration of cellulose-containing natural materials has been suggested in recent years as a method of determining the alpha-cellulose component of these materials (*4–7*). It has been also claimed that by this method a true value of the degree of polymerization of the undegraded alpha-cellulose fraction can be obtained. The most suitable nitrating medium for this purpose is the one applied by Alexander and Mitchell (*1*), containing nitric acid, phosphoric acid, and phosphorus pentoxide. Apart from the composition of the nitrating medium the other variables in this process are the time and temperature of nitration. The effect of the time of nitration on the yield of nitrated material, degree of polymerization and yield of nitrated alpha-cellulose have been studied in many cases, mostly for the purpose of finding the optimum conditions of nitration of a particular system. However, different workers applied different temperatures in the nitration stage. Alexander and Mitchell recommend a temperature of 20 ± 0.1°C. (*1*), but Mitchell, in an earlier work (*2*), nitrated at 28°C. Heuser and Jorgensen (*3*) nitrated spruce and aspenwood at a not carefully controlled temperature varying between 20 to 25°C. Timell (*4–7*) has applied different temperatures for different materials, e.g., cotton, flax, and ramie were nitrated at +4°C., milkweed at +16°C., hemp, jute, kenaf, and various species of wood at + 17°C.

In an earlier work (*8*) the results of the nitration of the woods of *Eucalyptus rostrata* (river red gum) and *Pinus halepensis* (Aleppo pine) at 20°C. were reported. Other nitrations carried out subsequently at lower temperatures, gave widely different results for the yield and degree of polymerization of the alpha-cellulose component. In the present work the results of a series of nitrations of the wood of *Eucalyptus rostrata* carried out at 3°C. and various times are reported, and the influence of these factors on the reactions involved discussed.

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EXPERIMENTAL METHODS AND RESULTS

Methods of preparation of samples, extraction of the acetone soluble fraction, determination of nitrogen content and degree of polymerization were described in detail in an earlier work (*8*). In the present work the flasks containing the nitrating medium and wood powder were placed in a refrigerator in which the temperature was maintained at 3°C., for lengths of time varying from 0.5 to over 1000 hr. The results are shown in Table I. All nitrations were carried out on 1-g. samples of dry wood powder. The notation of the various fractions is as follows: A, the crude nitrate; Al, the alpha-cellulose component, obtained by acetone extraction of fraction A and precipitating by pouring the extract into water; A2, the acetone insoluble fraction and A3, the acetone and water soluble fraction. Nitrogen contents and degrees of polymerization are of fraction Al.

DISCUSSION

In Figs. 1 (a–d) a graphical representation of the nitration process is given. These figures show the results of the nitration over the whole time range studied, and in addition the corresponding results obtained previously (*8*) at 20°C. are shown for comparison.

The yield of crude nitrate, fraction A (See Fig. 1a) is high at the initial period of the nitration process and reaches almost the value calculated viz. 1.49 g. (*8*). These early points confirm, therefore, the previously drawn curve for 20°C., where it was assumed that full nitration of all the wood components takes place instantaneously. A similar value for the yield of western red cedar wood nitrated in the vapor phase, where none of the components was dissolved, was obtained

Table I. Nitration of *Eucalyptus rostrata* Wood at 3°C.

Time o Nitration, hr.	*Fraction A. g.*	*Fraction A1. g.*	*Fraction A2. g.*	*Fraction A3. g.*	*Nitrogen of A1. %*	*Degree of polymerization of A1*
0.5	1.30	0.04	1.16	0.10	..	1250
1	1.31	0.07	1.10	0.14	..	1750
2	1.25	0.14	0.95	0.16	12.8	2000
5	1.05	0.26	0.65	0.16	13.0	2300
10	0.95	0.28	0.49	0.18	13.3	2400
24	0.93	0.29	0.48	0.16	13.8	2700
72	0.88	0.33	0.45	0.10	13.8	2500
120	0.86	0.36	0.41	0.09	13.7	2400
572	0.83	0.52	0.27	0.04	13.7	2000
1024	0.82	0.57	0.21	0.04	13.6	1950

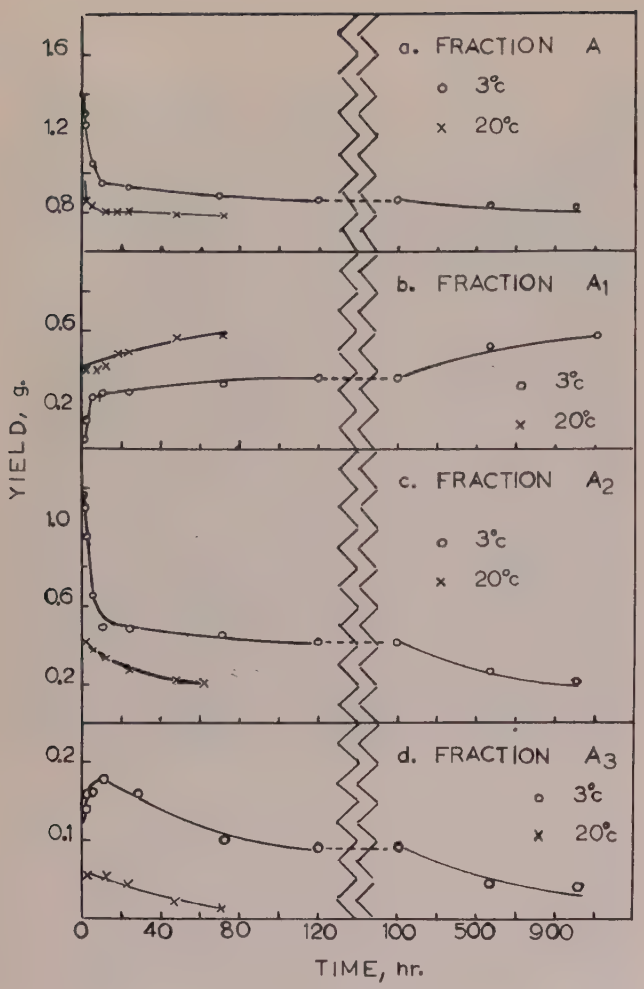

Fig. 1 (a–d). Effect of time of nitration on the yield of various fractions

by Elias and Hayward (*9*). The initial and rapid fall in the yield of this fraction is due to decomposition and solution of lignin nitration products. This process involves the breaking of lignin-carbohydrate bonds, which may be assumed to take place more slowly at the lower temperature, thus causing a slower rate of decrease in the yield of this fraction. As in the nitration at 20°C., the slow subsequent decrease in the yield of crude nitrate is due to dissolution of remnants of nitrated lignin as well as of carbohydrates.

The variations in the yields of fractions Al and A2, shown in Figs. 1b and 1c are, as in the case of the nitrations at the higher temperature, of a complementary nature. A rapid reaction is seen to take place in the first 10 hr. of nitration at 3°C. and it may be assumed that at 20°C. this reaction was complete after 2 hr. of nitration and was therefore not detected. If it is assumed, as suggested earlier (*8*), that these changes are due to the breaking of a bond between the alpha-cellulose and hemicellulose components of the wood then it appears that the decomposition of the alpha-cellulose-hemicellulose complex takes place in two stages. The first and rapid stage takes 10 hr. at 3°C. and less than 2 hr. at 20°C. The second and slower stage proceeds at a lower rate at the lower temperature, so that the levels of these fractions reached after 72 hr. of nitration at 20°C. are reached at 3°C. only after 1000 hr. If, as suggested previously (*8*) the bond between the alpha-cellulose and hemicellulose is an ester bond between a hemicellulose carboxyl group and a cellulose hydroxyl group, then the existence of two types of links may be due to the points of attachment being partly at primary and partly at secondary hydroxyl groups. It has been shown by Purves and his co-workers (*10, 11*) that primary and secondary hydroxyl groups of cellulose react at different rates, the former more rapidly. It may be, therefore, assumed that the initial rapid reaction corresponds to the breaking of a bond between a hemicellulose carboxyl and a primary hydroxyl of cellulose, whereas the slower reaction corresponds to the breaking of a similar bond formed at a secondary hydroxyl group. A similar effect was found by Hiller (*12*) in the homogeneous acetylation and deacetylation of cellulose. The second stages of the process are plotted separately as log plots in Fig. 2. These plots are linear and enable the calculation of first order reaction constants at the two temperatures and also an approximate value for the energy of activation. The following values were obtained:

$k_{20} - = 3.21 \times 10^{-4}$ min.$^{-1}$
$k_3 - = 2.76 \times 10^{-5}$ min.$^{-1}$
Ratio $k_{20}/k_3 = 11.6$
$E = 23$K cal.

The value of 23Kcal. is normal for homogeneous reactions taking place at ordinary temperatures (*13*). However, very little is known about heterogeneous reactions of this nature. Values of the activation energies of homogeneous acid hydrolysis of esters vary between 20 and 30 Kcal. per mole. Hiller (*12*), in a study of the system cellulose–acetic acid–water, obtained values of 20.6 ± 0.4 and 8.5 ± 0.2 Kcal. per mole for the activation energy of the homogeneous uncatalyzed and acid catalyzed deacetylation of the secondary hydroxyl groups of cellulose respectively, at a temperature range of 40 to 100°C.

A possible mechanism for the acidolysis of the complex is as follows:

cellulose—O—CO—hemicellulose + H_2NO_3 + → cellulose—O—NO_2 + HOOC—hemicellulose + H^+

The H^+ is furnished by the phosphoric acid which plays the role of the acid catalyst. The complex ion $H_2NO_3^+$ may be the hydrated form of the ion NO_2^+ which is assumed to be the active agent in the nitrating medium (*14, 15*).

The mode of variation of the amount of fraction A3, which consists mainly of nitrated hemicellulose, with the time of nitration, shown in Fig. 1d, may also be ex-

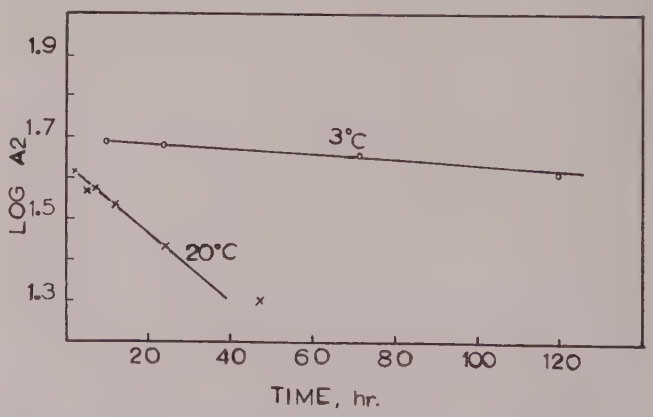

Fig. 2. Effect of time of nitration on the log yield of fraction A2

plained in terms of the suggested complex. The rapid initial increase in the amount of this fraction is due to the release of the hemicellulose from the complex, whereas the consequent decrease is due to the decomposition and solution of these carbohydrates by the nitrating acids.

In the previous work (*8*) it was suggested that the abrupt fall in the degree of polymerization of fraction A1 observed after 20 hr. of nitration at 20°C. may be due to one of two reasons: the sudden release of a large number of shorter cellulose chains and/or the depolymerization of the cellulose at the points where the hemicellulose chain was linked to the alpha-cellulose. From the results described above it seems that the second possibility is more likely. In the nitration at the lower temperature only 0.04 g. of fraction A1 are obtained after 0.5 hr. of nitration, i. e., the process of the breaking of the hemi-alpha complex is still at its beginning and may be followed more closely. If it is assumed that the hemicellulose-alpha-cellulose bonds are randomly distributed, then the shorter chains of the cellulose nitrate will have a smaller number of links per molecule and will therefore be the first to dissolve. Thus, as can be seen in Fig. 3, relatively low degrees of polymerization of 1250 are observed for the A1 fraction obtained in the early stages of the nitration. The value increases rapidly as the number of bonds broken increases with the time of nitration. In the nitration at 20°C. the majority of the complex is destroyed in the first 2 hr. of nitration, and hence this effect is not noticed. The nitrating acids also take part in a depolymerization reaction occurring preferably at the points of attachment of the hemicellulose residue to the alpha-cellulose. It may be pointed out that chain branches are among the possible naturally occurring structural abnormalities which have been proposed as sites of weak links in cellulose (*16*).

It is interesting to note (see Fig. 3) that in the time interval of 20 to 120 hr. of nitration the degree of polymerization values for both temperatures fall almost on the same curve. This may have suggested that the depolymerization of alpha-cellulose by the nitrating acids proceeds at the same rate both at 3°C. and at

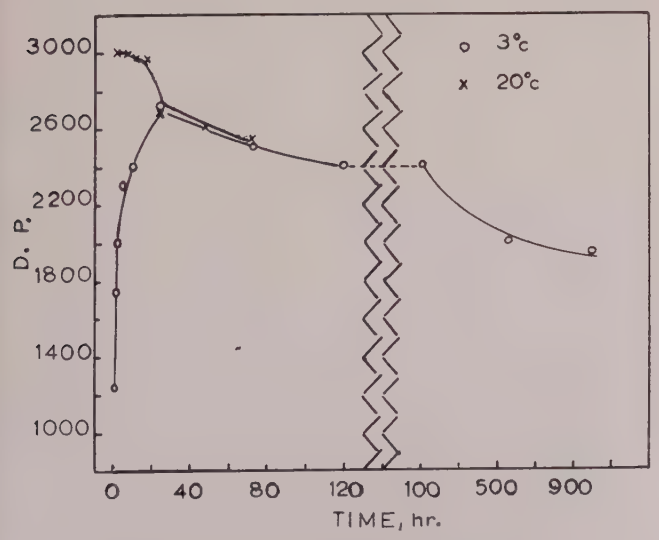

Fig. 3. Effect of time of nitration on the degree of polymerization of alpha-cellulose (fraction A1)

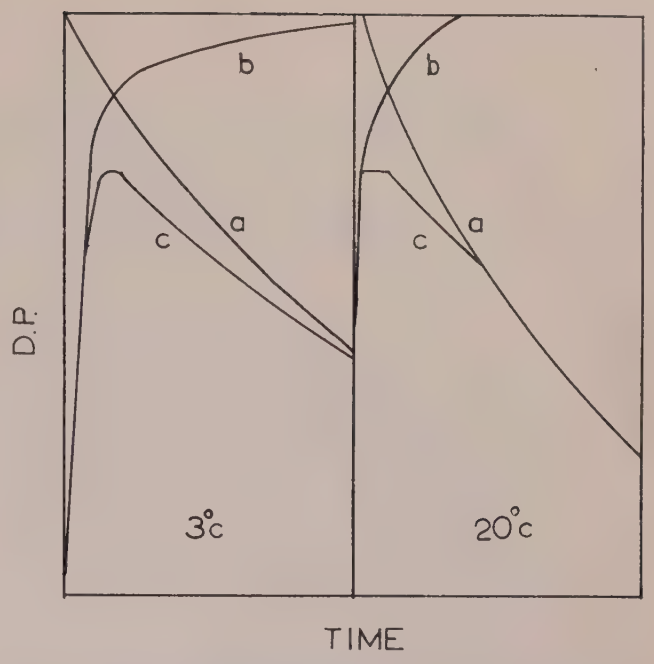

Fig. 4. Schematic representation of the variation of the degree of polymerization of alpha-cellulose (fraction A1) with time of nitration

a—Effect of depolymerization
b—Effect of decomposition of complex
c—Resultant

20°C., i. e., it is not temperature dependent in this range of temperatures. However, these seemingly equal rates of depolymerization are coincidental. The values of the degree of polymerization obtained at 20°C. after 24 hr. of nitration refer to a higher yield of alpha-cellulose (27.4%, see Table II) and consequently to a greater number of longer chains released from the complex, as indicated above. The corresponding value at 3°C., however, refers to the much lower yield of alpha-cellulose, i. e., 16.2%, with a corresponding smaller number of longer chains. If no depolymerization had taken place, or if the rates of depolymerization at the two temperatures were equal, a higher value of the degree of polymerization would have been expected for the alpha-cellulose of the 20°C. nitration after 24 hr. than the corresponding value at 3°C. Since these values are close it is evident that the depolymerization at 20°C. proceeds at a higher rate than at 3°C.

In Fig. 4 a schematic representation of the two main simultaneous reactions affecting the degree of polymerization of the alpha-cellulose is drawn for both temperatures studied. The a curves correspond to the depolymerization reaction, and the b curves correspond to the effect of the decomposition of the complex on the degree of polymerization. The c curves, obtained by combination of the two curves a and b, are seen to resemble in shape the experimental curves shown in Fig. 3.

The results reported in this paper do not enable the determination of the temperature dependance of the depolymerization reactions which take place during the direct nitration procedure, to any degree of accuracy. It is evident, however, that the decomposition of the alpha-hemi complex is more strongly influenced by the

temperature than the depolymerization reaction. After 1024 hr. at 3°C. and 72 hr. at 20°C., 32.2% and 31.7% of alpha-cellulose have been released, respectively (see Table II), from the alpha-hemi complex. However, the degree of polymerization values are respectively 1950 and 2540. If the temperature dependance of the depolymerization reaction were similar to that of the decomposition of the complex, the latter values should have been similar.

It is seen that the values of the alpha-cellulose obtained by the method of direct nitration are strongly dependent on the time and temperature of nitration. This dependance is different for various kinds of lignocellulosic materials, as evident from the results obtained with *Eucalyptus rostrata* as compared to those of *Pinus halepensis* (*8*). It is difficult therefore to ascribe a definite significance to any alpha value obtained, unless the exact conditions of the nitration are specified and unless the behavior of the given lignocellulosic material and of its possible alpha-cellulose-hemicellulose complex are known. The complicated nature of the reaction involved does not therefore enable the determination of the alpha-cellulose content with any greater accuracy than the alkali method.

Table II. Yields of Apparent Alpha-Cellulose

Temperature of nitration, °C.	*Hours of nitration*	*% Alpha-cellulose*	*Degree of polymerization*
3	2	8.3	2000
	5	15.2	2300
	10	16.0	2400
	24	16.2	2700
	72	18.4	2500
	120	22.0	2400
	572	29.2	2000
	1024	32.2	1950
20	2	23.2	3000
	12	23.8	2960
	24	27.4	2680
	48	31.3	2600
	72	31.7	2540

Analytical alpha 43.1 determined by TAPPI Standard T 203 m 55 on chlorite holocellulose and corrected for ash, uronic anhydride, and pentosans.

In Table II the values of the apparent alpha-cellulose calculated from the nitrates are presented. It is seen that these values vary widely with the time and temperature of nitration and are considerably lower than the value of the "true" alpha-cellulose obtained by the alkali method from the chlorite holocellulose and corrected for ash, uronic anhydride, and pentosans.

Similar considerations apply to the D. P. determination by the nitration procedure. During the nitration a depolymerization occurs. Additional depolymerization may take place on the first instant of nitration when almost all the hydroxyl groups are nitrated. This initial depolymerization cannot, however, be accurately determined since the alpha-cellulose-hemicellulose complex decomposes gradually only later. Since the shorter cellulose chains will be the first to be liberated, abnormally low values will be obtained. Furthermore, the fractionation of such nitrated alpha-cellulose will not provide any significant information on the chain-length distribution of the cellulose.

It is evident, therefore, that any determination of the chain-length distribution of the alpha-cellulose obtained by the direct nitration of the wood of *Eucalyptus rostrata* will be misleading. If such a fractionation is carried out on the products of the nitration at 20°C., a certain amount of alpha-cellulose will be missing from the crude nitrate, fraction A, already after 2 hr. of nitration (*8*). A considerable part of the alpha-cellulose contained in fraction A will not dissolve in acetone before the complete decomposition of the alpha-hemi complex. If, however, the fractionation is carried out on a product nitrated for a time sufficiently long to decompose the complex, the results will again not represent the original chain length distribution of the cellulose in the wood since during the nitration a slow but marked depolymerization takes place. As seen above, decreasing the temperature from 20°C. to 3°C. does not eliminate the depolymerization effect.

While the direct nitration procedure does not seem to be particularly suitable for accurate analytical determination of the alpha-cellulose and its D. P. distribution, some interesting information may be obtained by this method on the state in which the alpha-cellulose exists in the native ligno-cellulosic material, and the differences in the nature of the alpha-cellulose-hemicellulose complex may serve as a characterization of a given lignocellulosic material.

CONCLUSIONS

1. In the case of *Eucalyptus rostrata*, the direct nitration of the wood comprises the following reactions: (a) instantaneous nitration of all wood constituents, (b) the cleavage of the alpha-cellulose-hemicellulose bond. (c) the decomposition and dissolution of the nitrated lignin and some carbohydrate portions, and (d) the depolymerization of the alpha-cellulose.

2. The cleavage of the alpha-cellulose-hemicellulose bond is temperature dependent with E-23 Kcal. per mole. The depolymerization of alpha-cellulose by the nitrating mixture is slower at 3°C. and more rapid at 20°C.

3. The degree of polymerization of the alpha-cellulose increases rapidly with time of nitration, reaches a maximum and then falls off slowly. This is due to the combined effect of 1(b) and 1(d).

4. The direct nitration of the wood of *Eucalyptus rostrata* (river red gum) is not a suitable method for the analytical determination and separation of alpha-cellulose, for the degree of polymerization of the cellulose in the native state and for its distribution.

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A Nonhydrolytic Spectrophotometric Method for Determining Lignin in High-Purity Pulps

RICHARD BARTUNEK

The intensity of the yellow color developed by treating wood pulp with warm dilute nitric acid is used as the basis for determining "lignin number." During the past 33 years, the decrease in consumption of viscose filter material has closely paralleled the decline in the lignin number of dissolving pulps. Ultraviolet spectrophotometric investigations, on nitric acid extracts from various pulps, showed that extinction maxima at 261 and 330 to 338 mμ were pronounced enough to allow differentiation between todays high-purity chemical pulps. It has been established that these maxima have their origin with the structural units of the lignin; source of the 261 mμ maximum is the benzene ring, while the 330 to 338 mμ maximum originates with the phenolic hydroxyl groups. Neither hemicelluloses nor extractives noticeably interfere.

ONE of the most difficult and controversial problems in the analysis of wood and wood pulps is the determination of lignin in the raw materials, intermediates, and end products. The usual method consists in removing all accompanying substances, the final residue being referred to as lignin. Resinous, waxy, and fatty substances are removed by solvent extraction while carbohydrates are removed by hydrolysis. This method can hardly be considered an absolute one, since the carbohydrate content of the residue is unknown; and some proportion of the original material may have become acid insoluble through conversion to humus. Recent investigations, such as the one by Kuerschner (*1*) with alcoholic nitric acid, have questioned the correctness of lignin analyses on wood and on raw pulps.

All methods of lignin determination fail almost completely if applied to bleached pulps or to modern highly purified chemical pulps. The common dyeing methods, e.g., with Malachite Green, indicate that even chemical pulps contain traces of lignified fiber particles, but these methods are unsuitable for quantitative purposes.

However, for over 50 years there has existed a method for characterizing the lignin content of vegetable raw materials and even of fully bleached surgical cotton. Although exceptionally simple, quick, and reproducible, this method has found little application in industrial practice, and none in purely scientific areas. We refer to the reaction of 13% HNO_3 at 40°C. with all vegetable raw materials and pulps. The nitric acid develops a yellow color, and is reduced to nitrous acid (HNO_2) and nitric oxide (NO); in the presence of atmospheric oxygen, the latter is immediately oxidized to nitrogen dioxide. The first to utilize these reactions for determining the degree of purity of cellulose was Seidel (*2*), working under Hempel at the Dresden Technical Institute. In this work, most importance was attached to the rather complicated analysis of the evolved gases. Knowing of Seidel's work E. Richter (*3*) at Hawkesbury then developed an industrially applicable method for bleachability, based on the yellow coloration of 13% HNO_3. A standard pulp was used for comparison.

In 1926, when we had to explain the very low filterability of a shipment of viscose pulp, we went back to this almost forgotten nitric acid method and found it so suitable that we have since been using it most successfully for evaluating the processibility of viscose pulps. We found that a suitable stable colorimetric standard is 0.01% $K_2Cr_2O_7$ solution which has been given a slightly reddish tinge by the addition of a little potassium permanganate.

The working method is very simple: 15 g. of pulp, calculated on a moisture-free basis, is cut into 5 mm. squares, and placed in a glass-stoppered flask containing 100 ml. of 13% HNO_3 at 40°C. After 45 min. at this temperature, the mixture is filtered through sintered glass, the yellowish filtrate cooled rapidly and immediately analyzed colorimetrically. The comparison standard in prepared by dissolving 0.100 g. $K_2Cr_2O_7$, in water which has been distilled over permanganate and then adding 0.2 ml. 0.1 *N* $KMnO_4$; the mixture is finally diluted to 1000 ml. If the colors of the nitric acid extract and the standard solution are the same, the lignin number of the pulp is 1.0. This corresponds to an unbleached rayon pulp containing about 1% lignin by the acid hydrolysis method. The lignin numbers of modern rayon pulps are between 0.01 and 0.02; these values are unaffected by the presence in the sample of hemicellulose or extractives.

Figure 1 illustrates the changes in filterability and lignin number which have occurred in spruce sulphite viscose pulps during the last 30 years. The scale at the left is lignin number while the scale at the right represents filterability in terms of "filter factor";

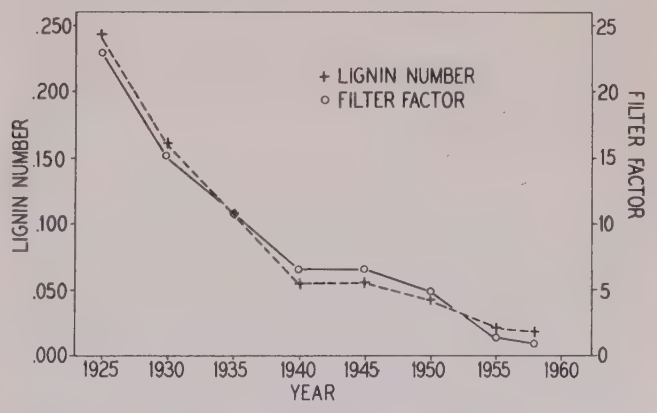

Fig. 1. Lignin numbers and filter factors for spruce pulps, 1925–58

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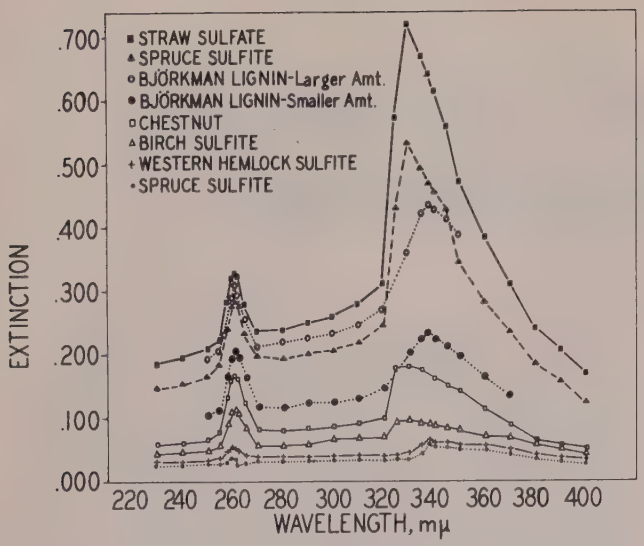

Fig. 2. Extinction curves of nitric acid extracts from different types of pulp

this latter is a simple way of recording filtration data in plant-scale operations and is the number of square meters of filter clothing consumed in filtering 10,000 l. of viscose. The parallelism of the two relationships is rather impressive; the disturbance between 1935 and 1952 is attributed to the introduction of lower alkali viscoses. However, in recent years better dissolving techniques have compensated for lower alkali, and the original relationship between filterability and lignin number has re-established itself.

However, for today's pulps with lignin numbers of about 0.01, colorimetric methods based on visual evaluation of the yellow color are no longer sufficiently sensitive. The following possibilities existed for increasing the sensitivity:

1. Increased development of the yellow color by increased acid concentration, temperature, or reaction time.
2. Estimation of the yellow color with a photoelectric colorimeter, equipped with suitable filter.
3. Use of absorption or extinction photometry at suitable wavelengths in the ultraviolet.

We decided on the latter because we hoped this method would allow us not only to establish the lignin as the source of the yellow color, but also to gain further insight into the mechanism of the color development.

In the first series of investigations, extinction values for different types of pulp and for an extract of Björkman lignin were measured over the range 200 to 900

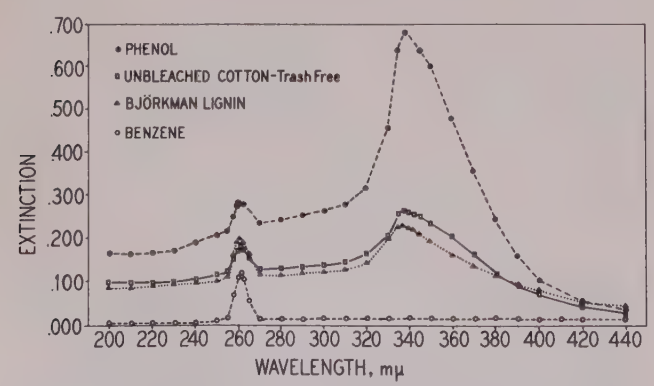

Fig. 3. Extinction curves with solutions of model substances in 13% nitric acid

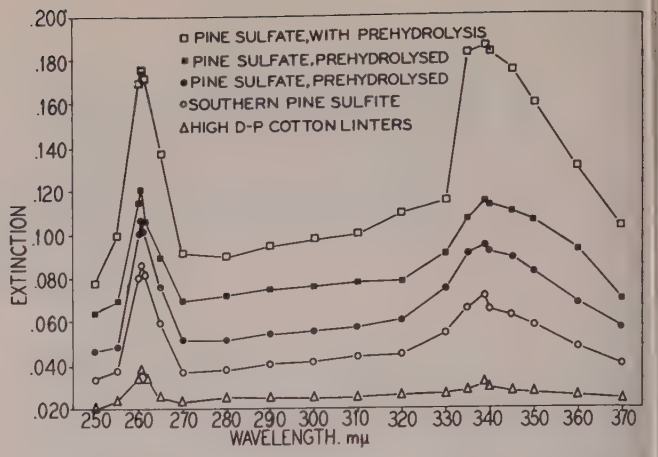

Fig. 4. Extinction curves for commercial tire cord pulps with higher lignin numbers

mmu, using a 10-mm. layer of solution. Björkman lignin is prepared by extraction of woodmeal with aqueous dioxane at 20°C., and is considered one of the least modified of the native lignin preparations.

Figure 2 shows the extinction curves in the interesting region between 230 and 400 mmu. These were made with a Beckman DU spectrophotometer using a hydrogen source below 325 mmu, and a tungsten lamp above 325 mmu; between 300 and 400 mmu a stray light filter was used.

The materials tested represent a considerable range of pulp types, and include spruce, birch, western hemlock, chestnut, straw, sulphite, and sulphate. The similarity in the shape of the curves is quite apparent; the extinction maximum at 261 mmu is especially pronounced, while at higher wavelengths the maximum varies between 330 and 338 mmu.

The two dotted curves were obtained from Björkman lignin, the upper with more lignin, the lower with less. They show maxima in the same region, although for these early samples a stray light filter was not used. When further supplies of Björkman lignin allow repeat testing with the proper filter, it is expected that the maxima will be somewhat shifted toward the shorter wavelengths. The occasional intersections of the Björkman lignin curves with the pulp lignin curves indicate structural changes of the residual pulp lignin during cooking and bleaching.

We next investigated which groups were involved in the formation of the two maxima: If lignin structural groups are involved, it would be the aromatic, and

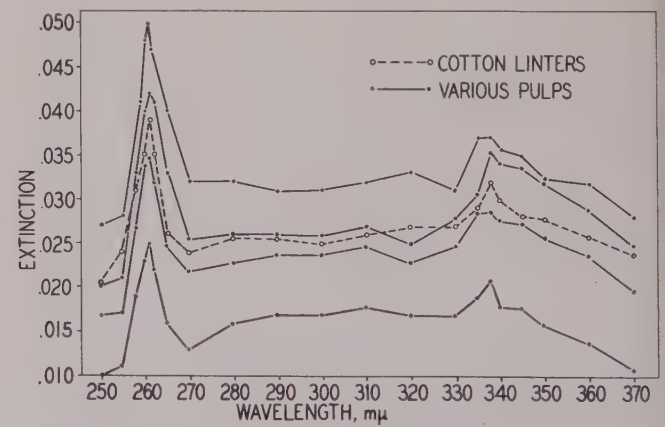

Fig. 5. Extinction curves from commercial spruce rayon pulps with low lignin numbers

particularly the phenolic groups which pass into the nitric acid. Consequently, our first step was to dissolve phenol in 13% HNO_3.

Figure 3 shows that the maxima obtained were at 261 and 338 mμ just as for Björkman lignin and the various pulps. The substances split off by nitric acid from both the Björkman lignin and the pulp lignin therefore contain phenolic groups. The dissolution of benzene in 13% HNO_3 (lower curve) showed that the 261 mμ maximum must be ascribed to the benzene ring, while the 330 to 338 mμ maximum is due to phenolic hydroxyl groups; both are probably in a slightly nitrated form.

The next two figures show the extinction curves for various dissolving pulps. Figure 4 illustrates a group of tire cord pulps, with lignin numbers ranging from 0.04 to 0.13, while the extinction maxima at 261 and 330 to 338 mμ range from 0.03 to 0.18. Figure 5 shows the curves for standard rayon pulps with lignin numbers between 0.01 and 0.03; here the 261 extinction maxima occur between 0.025 and 0.05, and the 338 maxima between 0.02 and 0.04. A high-D.P. linters pulp, represented by the dotted line, fitted in very well among the other pulps.

It was desired to establish a means of translating lignin number data from the modern Beckmann instrument back to the older well established visual colorimetric basis.

In Fig. 6 A extinction values are plotted against visual colorimetric lignin numbers for measurements made with three instruments: (1) Lange colorimeter with 420 mmu filter, (2) Zeiss Elko II with 420 mmu filter and (3) the Beckman DU (420 mmu) (Model 54700) (Lange colorimeter: line below.)

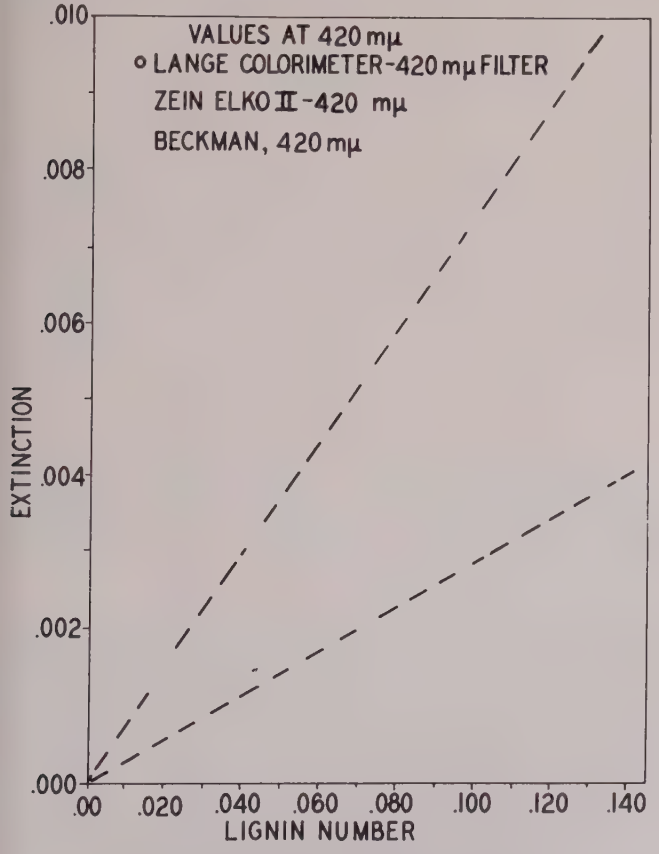

Fig. 6A. Relation between lignin numbers obtained by visual colorimetry and by extinction (Lange colorimeter: line below)

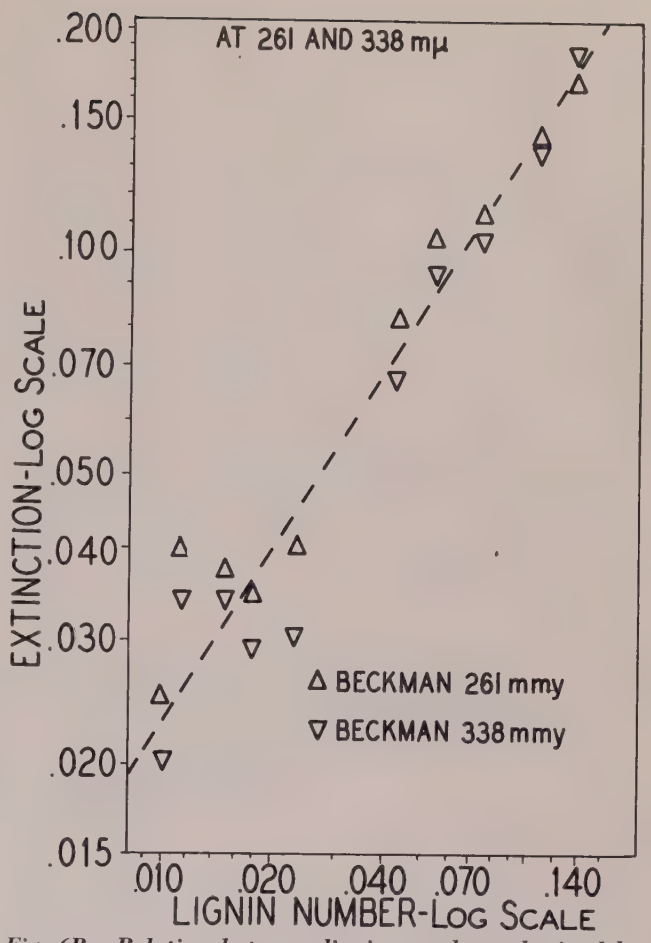

Fig. 6B. Relation between lignin numbers obtained by visual colorimetry and by extinction values

In Fig. 6 B is shown another graph of extinction values versus visual colorimetric lignin number; this graph, in which both scales are logarithmic, uses the Beckman extinction values at 261 and 338 mmu.

From the two graphs, it is concluded that (1) there exist approximately linear relationships between the visual lignin number values and the extinction at 420 mmu and (2) there is an approximately linear log-log relationship between visual lignin number values and the extinction maxima at 261 and 338 mmu.

It is apparent that several items require further investigation: (1) the significance of the maxima at 261 and 338 mmu, (2) the significance of the extinction at 420 mmu, (3) the relationship between (1) and (2), and (4) the reason for the spread of the second maximum over the range 330 to 338 mmu.

From a study of these areas, it is hoped to obtain information on the effect of pulp lignin content on viscose filterability; and on the extent to which pulp brightness will survive the viscose process to be reflected in whiteness of the regenerated fibers. In this connection, it will be of especial interest to compare chlorine dioxide bleaching with hypochlorite bleaching.

It is also noteworthy that cotton linters show a distinct lignin effect, hardly distinguishable from that of wood pulp (refer to the dotted curve in Fig. 5). To check the possibility that seed residues were responsible, samples of fine combed unbleached cotton tested after hand removal of impurities; the data in Fig. 3 show lignin numbers of 0.300 to 0.400, with two ultraviolet maxima in the expected locations. The presence of lignin in cotton fibers therefore seems

rather probable and not unexpected since the cotton hair grows directly on the lignin-rich seed.

Although the relation between pulp lignin content, color development in nitric acid, and the UV extinction maxima are quite apparent in a qualitative way, the quantitative relations are still obscure. For example, only about 10% of the Björkman lignin is soluble in 13% HNO_3, so we can hardly assume the solubility of the residual pulp-lignin exceeds this value. It has also been found that the lignin number of pulp increases on long storage; in the case of a spruce pulp stored 15 years, the lignin number increased by a factor of 11.6, from 0.08 to 0.93. During prolonged storage, additional color-forming groups had become soluble, as had possibly all the residual lignin. A related phenomenon is the increase in lignin number during aging and xanthating; tests on nitric acid extracts of the regenerated fiber show extinction values higher than those of the original pulp. This point still requires thorough investigation.

Work is now in progress on the possibility of completely dissolving the pulp lignin in the nitric acid by increasing time, temperature, or concentration.

The nitric acid extraction method offers new possibilities for the study of residual pulp lignin; in highly purified pulps it allows characterization of the ligneous residue, but is not yet capable of furnishing exact quantitative data. This method has the virtue of simplicity, and we hope it will be of assistance in the manufacture of high-purity pulps of excellent processing characteristics.

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Refining of Softwood and Hardwood Kraft Pulps Separately and as Mixtures

J. R. PECKHAM and M. N. MAY

Samples of two commercial bleached kraft pulps, one made from southern pine and one from gumwood, were refined in a Valley beater, separately and in two-component mixtures. The mixture level was maintained at 60% pine and 40% gum pulp, but the blends were made by (1) mixing the pulps before beating, (2) beating each pulp separately and mixing the two pulps at the same beating interval, and (3) beating each pulp separately and mixing the highly beaten stock samples of one kind with lightly beaten samples of the other. Samples taken at a number of beating intervals were formed into TAPPI Standard handsheets which were tested for bursting, tearing, and tensile strength and folding endurance. The results obtained indicated that some small strength advantage might be obtained from beating the two kinds of pulp separately and then blending them. Some further small increase in bursting strength might possibly be obtained by beating the gum to a somewhat lower freeness than the pine, so that the blend provides the desired freeness level. The optimum range of beating in this manner may be very narrow.

It has often been stated that paper furnishes made up of a blend of pulps having different fiber characteristics would be improved by refining the components separately rather than as a mixture. This study was designed to investigate the effects of separate versus combined refining of a mixture of bleached southern pine and bleached gum kraft pulps.

Commercially prepared bleached southern pine kraft and bleached gum kraft obtained in dry lap form were chosen for the investigation.

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Samples of the pulps were blended in the proportion of 60% softwood and 40% hardwood fiber and were refined in a Valley beater using a 5500-g. bedplate load and convenient test intervals. Additional samples of the pulps were refined separately in the same manner. Handsheets made at the several beating intervals were tested for basis weight, apparent density, bursting, tearing, and tensile strengths, and folding endurance. A third set of samples was beaten separately and sheets were made from the refined pulps at each beating interval, blended in the proportion of 60% softwood-40% hardwood fiber. The separate refining was again repeated, and this time the pulps were blended in the proportions described previously, but using the following mixture of samples:

1. Pine, 5 min. refining; gum, 60 min. refining
2. Pine, 15 min. refining; gum, 40 min. refining
3. Pine, 30 min. refining; gum, 20 min. refining
4. Pine, 50 min. refining; gum, 5 min. refining

Handsheets were again made and tested. All of the beating, handsheet making, and testing were carried out as specified in TAPPI Standards T 200 m-43 and T 205 m-53, unless otherwise described.

DISCUSSION OF RESULTS

The results of the tests performed on the handsheets made during the beater evaluation of the separate pulps are shown in Table I. Table II contains the data concerning the handsheets made from a mixture of pine and gum pulps where both components were beaten for similar lengths of time. Figures 1 to 4 show these data plotted against Canadian Standard freeness.

It will be noted that the pine pulp developed burst, tensile strength, and folding endurance at quite a steady rate, and that tearing strength decreased rapidly at

Table I. Physical Properties of Unblended Pulps

		(Pulp) Pine	Gum
Canadian Standard freeness, ml.	0[a]	735	710
	5	695	695
	15	600	660
	30	390	560
	50	170	370
	70	65	180
Basis weight (25 × 40—500), lb.	0	47.5	48.4
	5	46.3	46.1
	15	44.8	46.1
	30	47.5	46.8
	50	46.4	46.0
	70	48.5	45.4
Caliper, mils	0	4.7	5.0
	5	4.2	4.5
	15	3.6	4.3
	30	3.5	4.0
	50	3.3	3.6
	70	3.2	3.4
Apparent density	0	10.1	9.7
	5	11.0	10.2
	15	12.4	10.7
	30	13.6	11.7
	50	14.1	12.8
	70	15.2	13.4
Bursting strength, pt./100 lb.	0	38	8
	5	68	14
	15	93	27
	30	114	47
	50	123	68
	70	131	84
Tear factor	0	2.42	0.43
	5	2.01	0.67
	15	1.43	1.19
	30	1.28	1.45
	50	1.12	1.35
	70	0.99	1.28
Tensile strength, lb./in. (corrected to 45-lb. basis weight)	0	10.6	3.6
	5	16.2	5.9
	15	23.1	9.6
	30	28.8	14.2
	50	31.2	18.5
	70	34.0	22.8
Folding endurance (M.I.T. fold)	0	14	0
	5	99	1
	15	543	3
	30	968	18
	50	1449	76
	70	2086	254

[a] Beating time, min.

first and then more slowly. The gum pulp, on the other hand, displayed steadily increasing tearing strength until late in the beating cycle. Bursting and tensile strengths were low, and folding endurance was very poor.

When the values for these properties are shown at two freeness levels (Table III), it is noted that only in

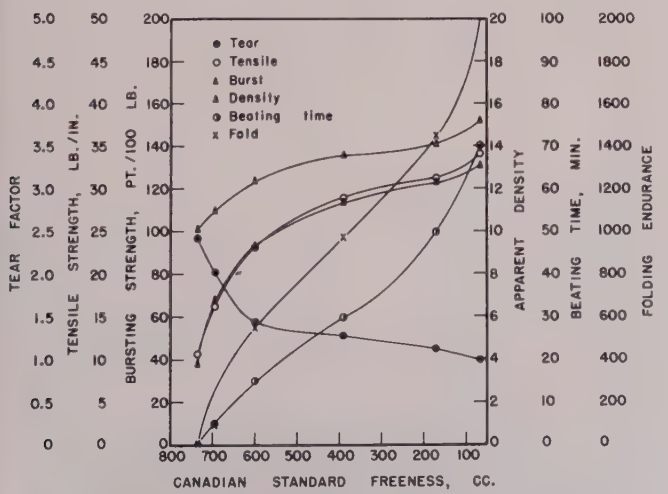

Fig. 1. Physical properties of bleached pine kraft pulp

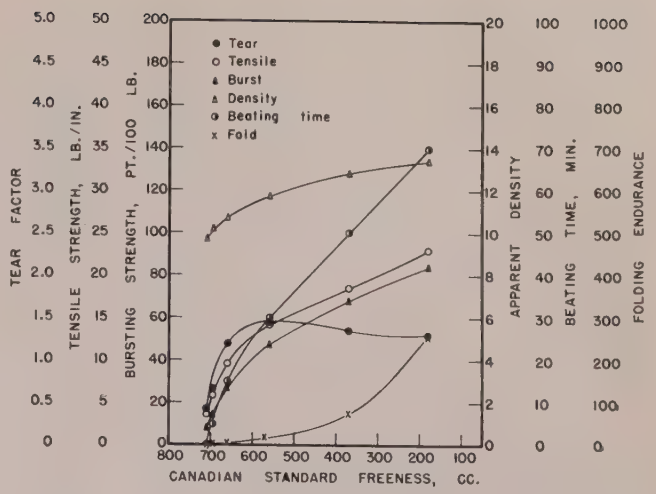

Fig. 2. Physical properties of bleached gum kraft pulp

tearing strength does the gum pulp strength approach that of the pine pulp.

In the same table, it can be seen that the strength of a pulp blend can be predicted very closely, if the two pulps are beaten before combining, simply by obtaining

Table II. Physical Properties of Pulp Mixtures

Pine, % → *Gum, % →*		*60* *40* Method of blending	*60* *40*
		Before beating	*After separate beating*
Canadian Standard freeness, ml.	0[a]	710	725
	5	695	710
	15	600	640
	30	400	485
	50	140	225
	70	...	90
Basis weight (25 × 40—500), lb.	0	47.7	45.6
	5	46.4	45.9
	15	46.4	42.0
	30	45.2	46.2
	50	46.9	46.9
	70	...	46.3
Caliper, mils	0	4.6	4.8
	5	4.2	4.4
	15	3.8	3.7
	30	3.4	3.6
	50	3.3	3.4
	70	...	3.1
Apparent density	0	10.4	9.5
	5	11.0	10.4
	15	12.2	11.4
	30	13.3	12.8
	50	14.2	13.8
	70	...	14.9
Bursting strength, pt./100 lb.	0	24	24
	5	42	41
	15	70	66
	30	92	89
	50	106	105
	70	...	114
Tear factor	0	1.74	1.75
	5	1.94	2.09
	15	1.53	1.62
	30	1.31	1.43
	50	1.19	1.26
	70	...	1.08
Tensile strength, lb./in. (corrected to 45-lb. basis wt.)	0	8.0	7.2
	5	12.2	12.2
	15	18.4	15.1
	30	22.4	23.3
	50	29.1	27.7
	70	...	28.8
Folding endurance (M.I.T. fold)	0	4	3
	5	16	16
	15	124	90
	30	440	348
	50	780	851
	70	...	1287

[a] Beating time, min.

Table III. Physical Properties of Pulps at Two Freeness Levels

	Composition of handsheets				
	Pine	*Gum*	*Pine gum blended before beating*	*Pine-blended after separate beating*	*Predicted values of pine-gum blends*[a]
Properties at 600-ml. Canadian Standard Freeness					
Beating time, min.	15	24	15	19	19
Apparent density	12.4	11.3	12.2	11.9	11.9
Bursting strength, pt./100 lb.	93	39	70	73	72
Tear factor	1.45	1.45	1.53	1.53	1.45
Tensile strength,[b] lb./in.	23.0	11.0	18.2	19.1	18.2
M.I.T. fold	540	13	125	140	329
Properties at 400-ml. Canadian Standard Freeness					
Beating time, min.	29	46	30	36	36
Apparent density	13.5	12.7	13.3	13.2	13.2
Bursting strength, pt./100 lb.	113	66	92	96	94
Tear factor	1.27	1.42	1.33	1.36	1.33
Tensile strength,[b] lb./in.	29.3	17.8	24.2	24.8	24.7
M.I.T. fold	960	65	440	510	601

[a] From weighted average.
[b] Corrected to 45-lb. basis weight.

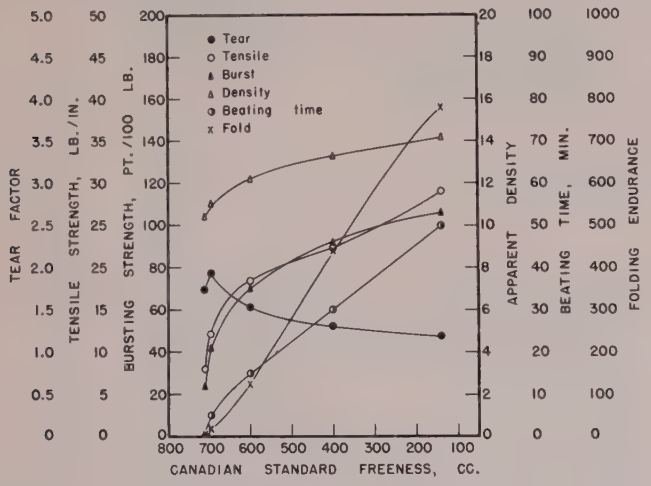

Fig. 3. Physical properties of a blend of pine and gum kraft pulps—beaten together

a weighted average of the strength properties at the two freeness levels. The notable exception to this is seen in folding endurance which was substantially lower for the

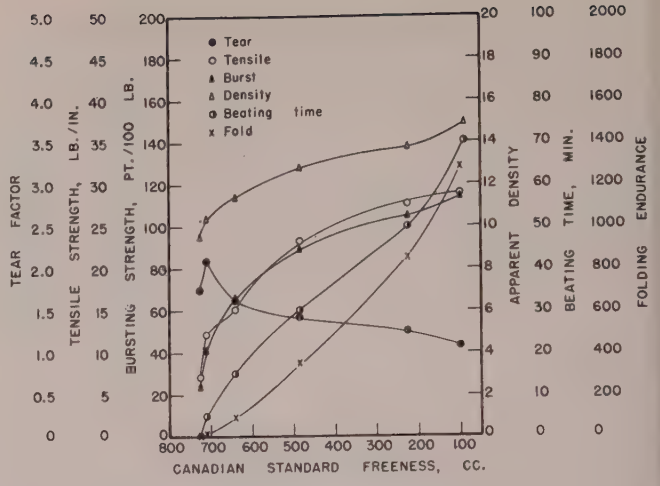

Fig. 4. Physical properties of a blend of pine and gum pulps—beaten separately

pulp mixture than would have been forecast. When the two pulps were combined and then beaten, the strength properties were slightly lower than when the components were beaten and then blended.

In the experiment where the two pulps were beaten separately and combined at freeness levels which differed substantially for each component, (see Table IV) it is unfortunate that the freeness measurements were, in error, recorded in Schopper-Riegler instead of Canadian Standard. Rather than use a conversion chart, the Canadian freeness data for previous runs were considered and the Canadian freeness values used in Table IV are the best possible estimates of the true levels. Figures in parentheses in this table are values taken from Fig. 4 at the freeness level which characterizes each blend. Thus, it will be seen that the mixture giving the best development of burst, tensile, and fold was pine beaten 15 min. and gum pulp beaten 40 min., and even that was only slightly stronger than would probably be found, were each pulp beaten to the freeness of the mixture and then combined. At all other levels of freeness the blends of pulps made at nonuniform freenesses resulted in a product inferior to blends made in other fashions. The fold values were particularly low for the mixtures made at dissimilar levels of freeness.

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Table IV. Physical Properties of Pulp Blends at Varying Freeness Levels

	Run			
	5A	*5B*	*5C*	*5D*
Pine, %	60	60	60	60
Pine beating time, min.	5	15	30	50
Pine freeness				
Can. Std. (est.), ml.	695	600	390	170
S.-R. (measured), ml.	875	845	750	410
Gum, %	40	40	40	40
Gum beating time, min.	60	40	20	5
Gum freeness				
Can. Std. (est.), ml.	260	460	625	700
S.-R. (measured), ml.	470	770	860	885
Mixture freeness				
Can. Std. (est.), ml.	520	545	485	380
S.-R. (measured), ml.	815	825	790	710
Basis weight (25 × 40—500), lb.	44.5	46.4	44.7	43.7
Caliper, mils	3.8	3.8	3.6	3.6
Apparent density[a]	11.7 (12.5)	12.2 (12.3)	12.4 (12.8)	12.1 (13.3)
Bursting strength, pt./100 lb.[a]	74 (85)	87 (81)	86 (89)	82 (98)
Tear factor[a]	1.64 (1.42)	1.47 (1.45)	1.43 (1.40)	1.42 (1.35)
Tensile strength, lb./in.[a] (corrected to 45-lb. basis wt.)	19.3 (23.0)	21.4 (21.2)	21.1 (23.0)	20.0 (25.2)
M.I.T. fold, double folds	132 (275)	215 (225)	201 (340)	201 (760)

[a] Figures in parentheses are interpolated values taken from Fig. 4 which characterize properties of beaten and blended mixtures at the Canadian Standard freeness level shown in each column.

Some Studies in Connection with the Spinning of High Tenacity Rayon from 100% Wood Pulp

BHARAT B. MITHEL, WILLIAM R. SAXTON, JAMES E. MORGAN, and JAN WITKAMP

Investigations were carried out on the influence of zinc sulphate in the spinbath, and modifier and sodium trithiocarbonate in the viscose, on the rate of neutralization and regeneration and water swelling. The modifiers studied were cyclohexylamine and Carbowax 1500 (polyethylene oxide—molecular weight 1500). The effect of modifiers on the regeneration rate of viscose was clearly shown by measuring the xanthate content of the partially regenerated yarn at various distances from the spinnerette. Modifiers retarded the initial regeneration, presumably due to a slower neutralization, but then regeneration became faster and after a certain point it was more complete, i.e., xanthate sulphur level of the yarn was lower than in yarns made from unmodified viscose. Zinc sulphate was shown to be a prerequisite for the action of modifiers. It was shown that even without modifiers in the viscose, zinc was effective in slowing the rate of regeneration; however it did not delay neutralization of the caustic. Sodium trithiocarbonate concentration in viscose was found to be critical and it affected decomposition and gel swelling significantly. Gel swelling of partially regenerated yarn was shown to be dependent on the state of regeneration, the highest values being obtained at the highest xanthate sulphur contents. Modifiers significantly lowered gel swelling for a given state of regeneration.

THE quality of high tenacity viscose rayons such as those used in the manufacture of automobile tires has improved very greatly in recent years. High tenacity rayon originated in 1935 when a patent granted to Givens, *et al.*, of Courtaulds (*1*) described a process for doubling the breaking strength of rayon. This process consisted of stretching the filaments in hot water or hot dilute acid while still in a partially regenerated state. They showed that regeneration could be suitably delayed by incorporating 4% $ZnSO_4$ in the acid spinbath.

In 1950 a patent was granted to Cox (*2*) of the DuPont Co. which showed that viscose with higher degrees of xanthate substitution could be spun by adding small amounts of monoamines to the viscose. This resulted in improved fine structure of the yarn and higher tenacities. Since then many of these "modifiers," as they are called, have been found and they produce yarns which are about 50 to 75% stronger than the older conventional tire yarns.

Apart from the patent literature, there are very few publications on the science and technology of high tenacity rayon. As a result of the trade secrecy maintained by the producers, there are many wide differences between commercial processes. A recent paper by Smith (*3*), however, discusses the steady improvements in quality of high tenacity rayon since 1940 due to the use of "modifiers."

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The object of this paper is to present data on the effects of some of the important factors involved in the manufacture of these modified or super tire yarns, in the hope that it will contribute to the understanding of the viscose spinning process. Studies were carried out on the effects of zinc concentration in the spinbath, and sodium trithiocarbonate and modifier in the viscose, on the rates of neutralization and regeneration of the viscose and the water swelling (degree of dehydration) of the resultant yarns.

In the spinning process for high tenacity rayons it is now generally accepted that the aim is to produce a large number of very small cellulose crystallites in a well ordered amorphous matrix. It is also desirable to have each cellulose chain passing through as many crystallites as possible (*4*). Measurements have shown a significant decrease in crystallite size in the modified rayons (*5*). These changes in fine structure undoubtedly occur in the early stages of spinning, when the viscose is extruded into the acid spinbath. It is with this end in view that some of the important changes which occur in the early stages of spinning have been investigated here.

Tire yarn processes differ in many ways from textile rayon processes. Higher quality cellulose pulps are used, they are xanthated to higher degrees of substitution, viscoses of lower cellulosic concentration and higher cellulose D.P. with higher ratios of caustic to cellulose concentration are prepared, and, of course, modifiers are now employed. Spinbaths are lower in sulphuric acid, higher in zinc sulphate concentrations, and are heated to higher temperatures. Sometimes modifiers are added to the bath in addition to the viscose.

During spinning three major changes take place. These are: (1) coagulation and dehydration, (2) neutralization of the alkali, and (3) dexanthation.

The three actions occur almost simultaneously. At first, viscose loses much of its water and the alkali is neutralized. The distance from the spinnerette which the thread must travel before neutrality is obtained is called the *D*-distance (*2*). Up to this point very little dexanthation occurs.

Gradually, as the acidity increases, dexanthation speeds up. This was followed by sampling the yarn at various immersions, quenching it immediately in a 0°C. buffer solution, freeing it from impurities, then estimating the xanthate sulphur content either spectrophotometrically or by iodimetric estimation of evolved carbon disulphide.

Swelling of the gel yarn was followed by the centrifugal method (*2, 6*).

METHODS AND PROCEDURES

Viscose. Natchez Tenacell N 157 HV (prehydro-

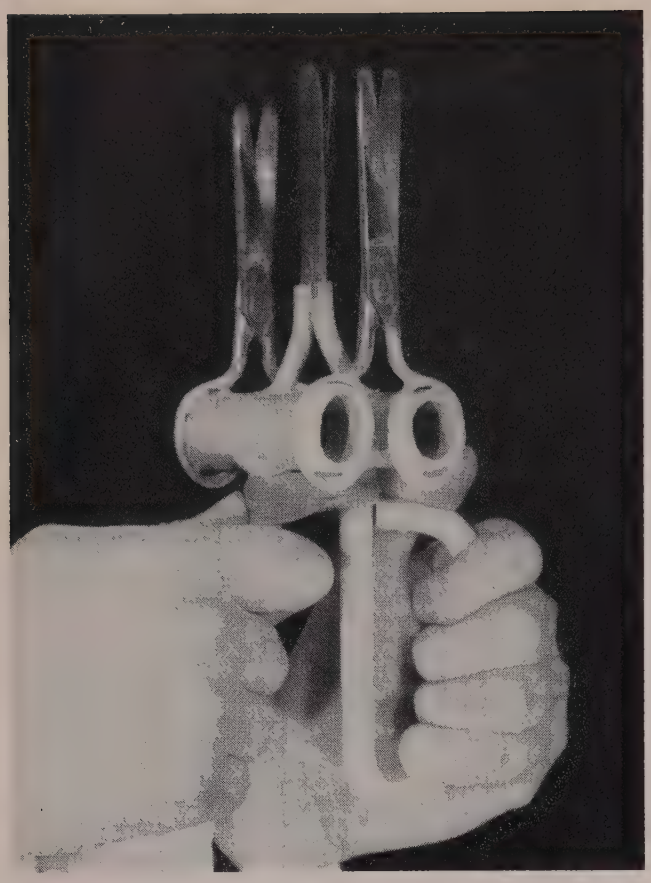

Fig. 1

lyzed sulphate) wood pulp was used in all experiments. The xanthates were prepared with 39% CS_2 on cellulose content of alkalicellulose. The viscose composition used was 6.25% cellulose and 5.75% NaOH and spinning viscosity was in the range of 60 to 70 poises. Viscose ripening was determined by the salt test and indices are expressed as percentage of sodium chloride solution.

Cyclohexylamine (C.H.A.) and polyoxyethylene glycol MW 1500 ("Carbowax 1500") were chosen as representative of the two main classes of "modifiers" in use today, viz., amines or polyoxyethylene glycol derivatives. The amounts added are expressed as a percentage of the cellulose in viscose.

Spinbath. The spinbath consisted of 90 g.p.l. H_2SO_4, 80 g.p.l. $ZnSO_4$, and 167 g.p.l. Na_2SO_4 (specific gravity 1.260 at 20°C.). Temperature was kept at 65±1°C.

Stretch Bath. The stretch bath consisted of 10 g.p.l. H_2SO_4, 10 g.p.l. $ZnSO_4$, and 20 g.p.l. Na_2SO_4 at 92±2°C.

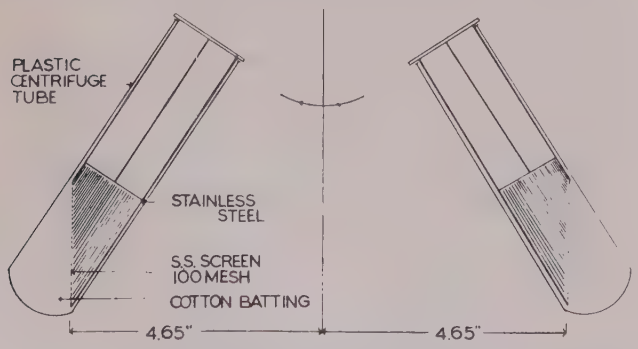

Fig. 2. Position of centrifuge tubes for gel swelling determinations

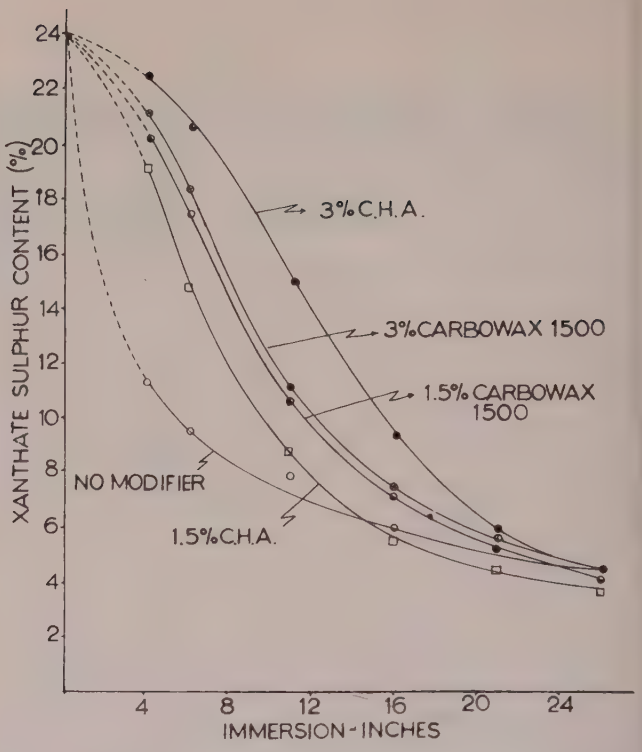

Fig. 3. Yarn xanthate sulphur contents at different immersions in the spinbath

Estimation of D-Value

The method of Cox (*2*) was used. This consists of mixing 0.1% bromcresol purple into the viscose and then observing the distance from the spinnerette at which the traveling filaments change from purple to yellow (pH 5.2 to 6.8). D-value measurements are accurate only to ±0.25 in. It was generally observed that this point was not very sharp, some filaments neutralizing faster than others.

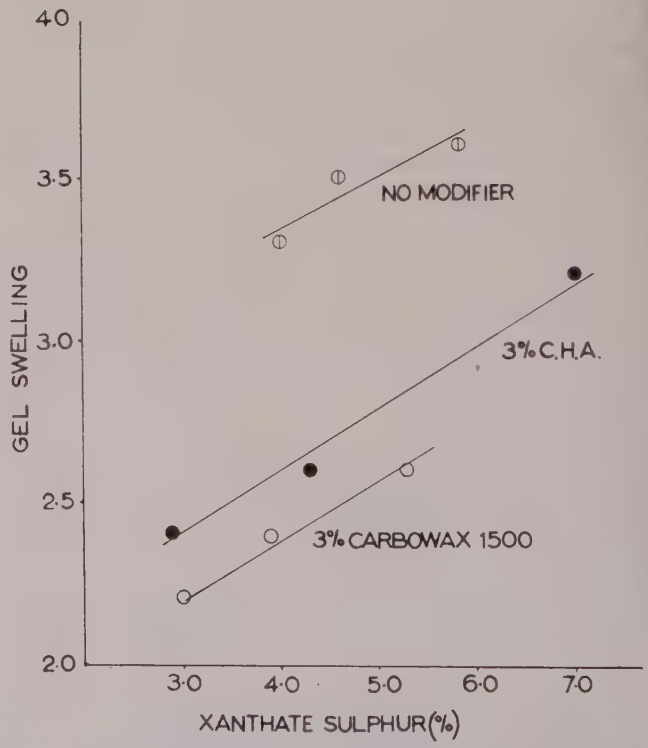

Fig. 4. Relationship between gel swelling and xanthate sulphur

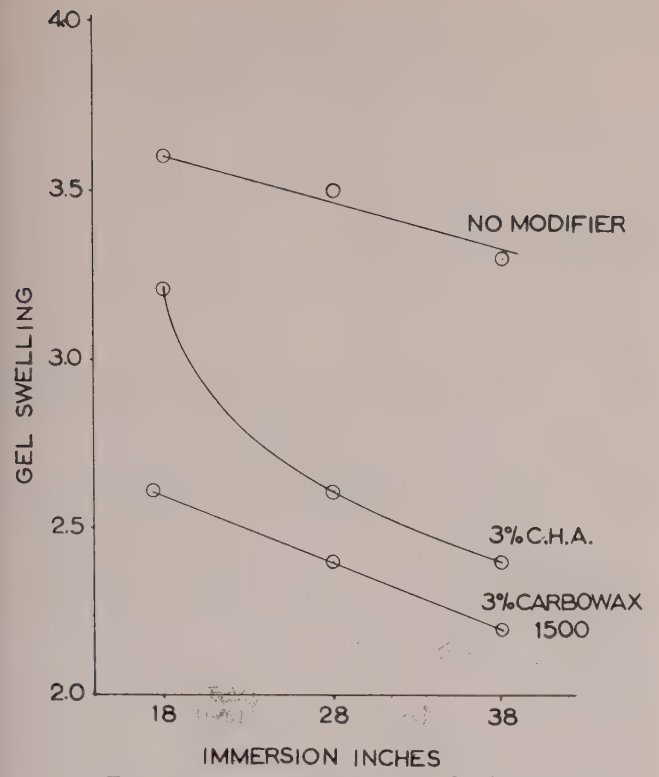

Fig. 5. Effect of immersion in spin bath on yarn gel swelling

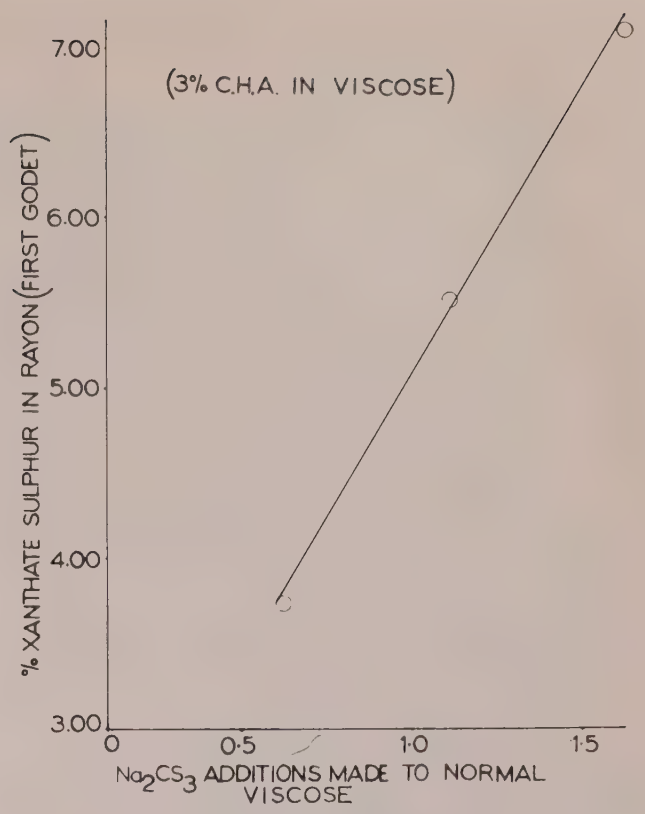

Fig. 7. Effect of trithiocarbonate content on rate of xanthate decomposition

Estimation of Xanthate Sulphur Content of Partially Regenerated Yarn

The yarn was sampled with the aid of a specially designed sampling device consisting of two pairs of scissors, the handles of which were connected together with spacers in such a way that a sample of yarn could be simultaneously cut and clamped. In most cases a 3-in. length was cut; however, to study short bath immersions a 1-in. length was used. The sampling device is shown in Fig. 1.

The yarn sample was immediately quenched in a stop bath of saturated sodium bicarbonate at 0°C. It was then thoroughly washed in a saturated solution of phosphate buffer at 0°C. having a pH of 6.5 followed by acetone to remove the remaining carbon disulphide and any modifier derivative that may have been present.

The sample freed from impurities was dissolved in 2 *N* caustic at −5°C. and then diluted to the proper concentration for spectrophotometric measurement of xanthate content at 303 mmu (*7*).

At very short immersions, however, the yarn contained too much unreacted viscose which dissolved if a washing procedure was performed. The yarn was neutralized in a 0°C. stop bath saturated with both sodium bicarbonate and sodium chloride to prevent sticking to the scissors. It was then dissolved in 2 *N* caustic and passed through an externally cooled anion exchange bed containing 10 g. of Amberlite IRA-400, and xanthate sulphur determined. The purified yarn solutions could be stored in a frozen state at −10°C. for up to 2 to 3 days without any appreciable decomposition of the xanthate.

Cellulose concentration of the caustic solution was calculated from viscose delivery, viscose composition, and withdrawal speed. A molar extinction coefficient value of 16,000 was used (*7*) and the xanthate sulphur

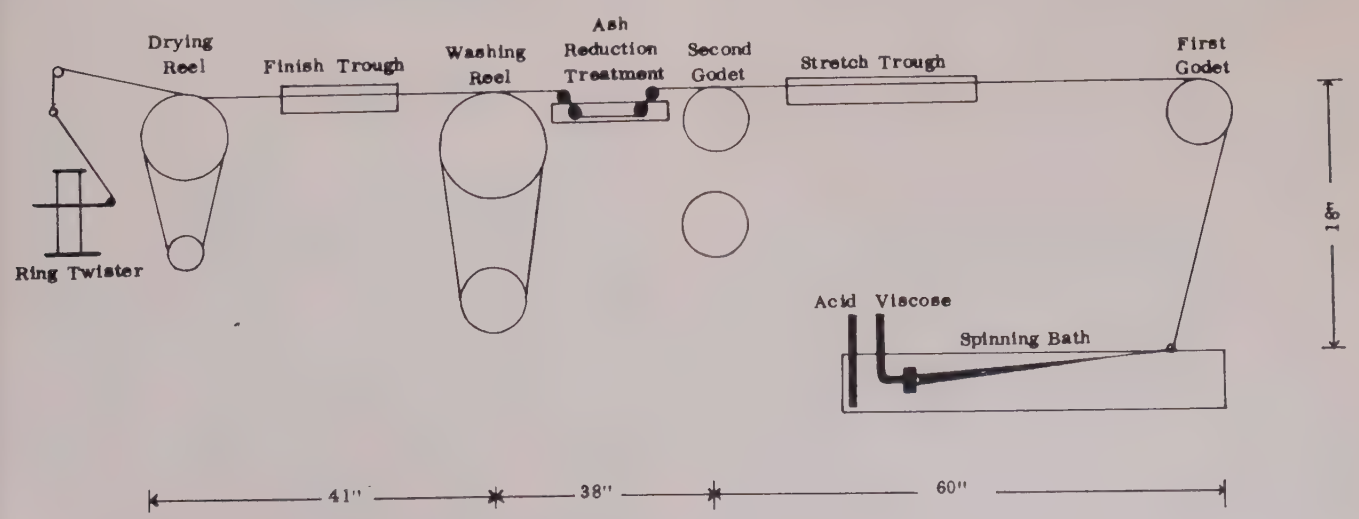

Fig. 6. Continuous spinning machine (not to scale)

was expressed as a percentage of the cellulose weight.

Estimation of Gel Swelling

Yarn samples were taken with the cutter described above, immediately quenched, and then left for 5 min. in a phosphate buffer solution at 0°C. having a pH of 6.5. They were then washed in 0°C. distilled water for another 5 min. and finally rinsed in a 1% Nacconol solution, also at 0°C. This rinse with wetting agent solution gave slightly lower but more reproducible results (*6*). The yarns were then centrifuged for 20 min. in closed plastic tubes with inclined perforated stainless steel screens at the bottom (Fig. 2). A Servall angle centrifuge (medium type SP provided with a W2 MT transformer) was operated at 4500 r.p.m., developing a relative centrifugal force of 2675 G.

The centrifuged yarns were immediately weighed, then allowed to dry overnight at 105°C. and reweighed. Gel swelling was calculated as the ratio of centrifuged to oven-dry weight.

The quenching bath and low wash water temperatures were unnecessary when determining primary and secondary swelling, i.e., swelling of fully regenerated undried and dried yarns, respectively.

RESULTS AND DISCUSSION

Spinning of Modified and Unmodified Viscose into Zinc and Zinc-Free Spinbaths

The results in Table I were obtained with 350 denier, 150 filament yarn instead of the 1650 denier, 720 filament commercial type yarn spun in most of the experiments. The sodium sulphate content was 167 g.p.l. for the zinc bath and 262 g.p.l. for the zinc-free bath in order to maintain a constant specific gravity.

The upper half of this table indicates clearly that modifiers delayed the neutralization and regeneration of the yarn. The delay in regeneration was more evident at 10 in. immersion. This means that while regeneration was at first delayed, it then proceeded at a faster rate in the case of the modified viscoses. Both modifiers accelerated the dehydration giving lower gel swelling.

Comparison of the results obtained with the two acid baths clearly shows that zinc sulphate was a prerequisite for the modifiers to act. Even without modifiers, zinc sulphate was effective in slowing the rate of regeneration. However, it had only a slight effect on D-value.

Effect of Modifiers on the Initial Rate of Regeneration of Viscose

In this series 1650 denier, 1080 filaments yarn was spun and 1-in. yarn samples were cut instead of 3-in. samples.

Table I. Effects of Modifiers and Zinc Sulphate on Partially Regenerated Yarn

Additions to viscose	*Zinc content in spinning acid, g.p.l.*	*Tests on partially regenerated yarn* — *D-value*	*Gel swelling at 20-in. immersion*	*% Xanthate sulfur* — *10-in. immersion*	*20-in. immersion*
None	80	2.5	4.67	6.2	4.3
3% Carbowax		4.0	4.01	9.4	5.0
3% C.H.A.		5.0	3.23	10.9	5.5
None		2.25	3.66	4.2	2.4
3% Carbowax	0	2.25	3.71	4.2	2.2
3% C.H.A.		2.0	3.77	3.7	2.1

Spinbath in each case contained 90 g.p.l. H_2SO_4 and had a specific gravity of 1.260 at 20°C.

Table II. Measurement of Yarn Xanthate Sulphur After Various Immersions

(Per cent sulphur on weight of cellulose)

Addition to viscose	*Immersion-distance from spinnerette, in.* — *4*	*6*	*11*	*16*	*21*	*26*
None	11.3	8.6	7.4	4.8	4.7	4.4
	11 5	9.8	8.8	6.0	4.8	4.6
1.5% C.H.A.	20.5	13.9	8.3	5.3	4.3	3.7
	17.4	15.5	8.8	5.9	4.6	3.9
3.0% C.H.A.	22.7	20.8	15.2	9.9	5.9	4.7
	20.4	19.7	15 0	9 3	5.9	4 5
1.5% Carbowax	20.8	17.7	10.0	7.4	5.7	4.0
	20.0	17.6	11.8	7.3	5.5	4.0
3.0% Carbowax	21.4	18.0	9.9	7.5	6.0	5.1
	20.8	18.4	13.0	7.1	5.4	4.2

The duplicate determinations were made on different days with different batches.

As explained above, the gelatinous yarn stuck to the scissors and tended to dissolve in the stop bath unless it was saturated with both sodium bicarbonate and sodium chloride.

Modified and unmodified viscoses were spun and yarn samples were taken after 4, 6, 11, 16, 21, and 26 in. immersion. Results are given in Table II. The duplicate measurements (made on different days with different batches) give some indication of the reproducibility obtained. The time between actual cutting and quenching was only about $^1/_{10}$ of a sec.; nevertheless at the speed at which the yarn was travelling this corresponds to almost 2 in. of bath travel. With this in mind the reproducibility may be considered satisfactory.

The results are plotted in Fig. 3. It is clear that each of the modified viscoses showed slower regeneration in the initial stages of immersion. Initial regeneration was slowest with 3% C.H.A. but after 26 in. immersion, xanthate sulphur level was similar to that of the other modifiers. The regeneration of the unmodified viscoses was rapid at first and then slowed down to a steady rate. It is obvious that the curves for all modified viscoses cross that of the unmodified. Evidence of this is shown in Table III.

The effect of concentration was much more pronounced with C.H.A. than with Carbowax; in fact there was almost negligible difference in the regeneration behavior between 1.5 and 3% Carbowax.

The following explanation is suggested to account for the above findings. When unmodified viscose is extruded from the spinnerette, the D-value is quickly reached and regeneration proceeds rapidly at first on the periphery of each filament. This results in a skin of regenerated cellulose through which acid must penetrate inwards to complete regeneration, and water and salt must diffuse outwards as the gel shrinks. This skin is probably not very permeable so that both processes of regeneration and gel shrinkage are slowed down. The results of the section following confirm that unmodified viscoses give higher gel swelling ratios.

When modified viscoses are extruded, perhaps a higher proportion of zinc xanthate is formed initially than with unmodified viscose. This is mere supposition and the mechanism by which the modifiers might

Table III. Effect of Acid Bath Immersion on Gel Swelling and Xanthate Sulphur

Acid bath immersion, in.	*No modifier* — *G.S.*	*X.S., %*	*3% C.H.A.* — *G.S.*	*X.S., %*	*3% Carbowax* — *G.S.*	*X.S., %*
18	3.6	5.8	3.2	7.0	2.6	5.3
28	3.5	4.6	2.6	4.3	2.4	3.9
38	3.3	4.0	2.4	2.9	2.2	3.0

Table IV. Water Swelling Measurements at Various Points in the Spinning Process

Sampling point	*Type of swelling*	*No modifier*	*3% C.H.A.*	*3% Carbowax*
Just before 1st godet	Gel	3.5	2.4	2.3
Just after 1st godet	Gel	3.2	2.2	2.1
At 2nd godet	Gel	2.41	1.85	1.84
End of washing	Primary	2.21	1.75	1.77
End of drying	Secondary	1.92	1.68	1.65

achieve this is not understood; however, work is going on at present in this laboratory to measure the proportion of xanthate in yarn which is combined with zinc. It is known (*9*) that zinc xanthate is more resistant to acid decomposition than is sodium xanthate. This outer membrane of more permeable zinc xanthate allows a better exchange of acid on one hand, and water and salts on the other. The section following confirms that for a given xanthate sulphur content in the partially regenerated yarn, gel swelling is lower than with unmodified viscoses, i.e., dehydration has been accelerated. Beyond 26 in. immersion, modifiers actually speed up regeneration.

Effect of Modifiers on Water Swelling of Tire Yarn

There are three types of water swelling used in characterizing tire yarn. Each is expressed as the ratio of wet centrifuged yarn weight to oven-dry yarn weight. Centrifugation must be performed under standardized conditions. The three types of swelling are:

Gel swelling (G.S.).....Applies to yarn which is only partially regenerated.
Primary swelling (P.S.).....Applies to fully regenerated yarn which has never been dried.
Secondary swelling (S.S.)....Applies to fully regenerated yarn which has been dried.

Initial experiments revealed, as expected, that gel swelling values were intimately associated with the xanthate sulphur content of the yarn. Length of immersion and bath temperature, for example, exerted pro-

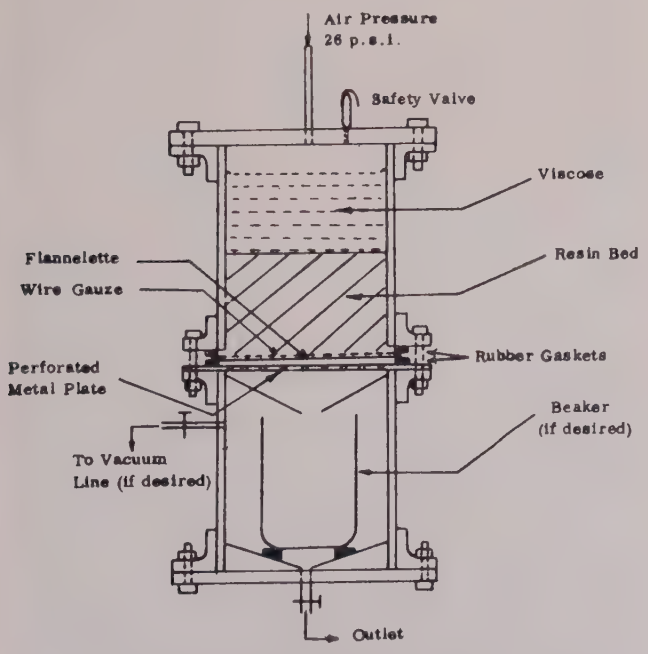

Fig. 8. Viscose purification apparatus (ion exchange)

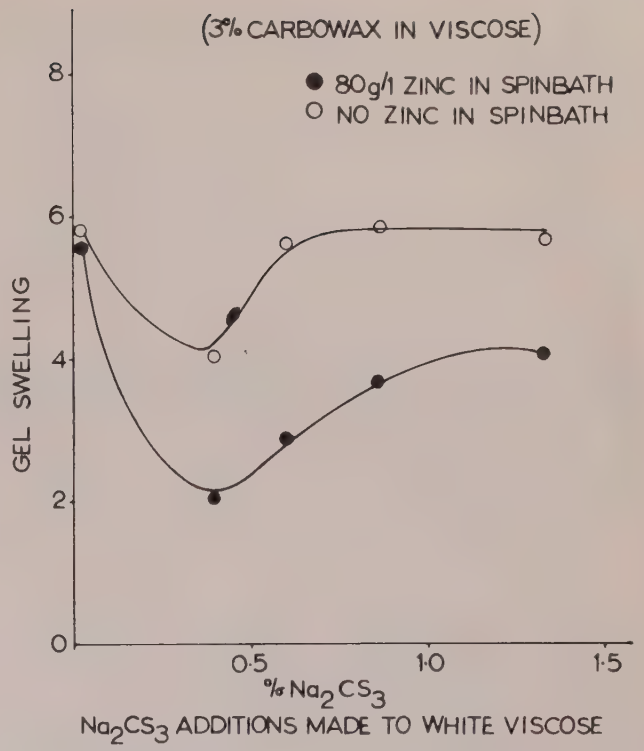

Fig. 9. Effect of trithiocarbonate concentration on gel swelling (20 in. immersion)

found effects. To study the relationship between xanthate sulphur and gel swelling, yarn samples were taken at various immersions and both tests were made. Viscoses, which were unmodified, and modified with 3% C.H.A. and 3% Carbowax, were compared. The results in Table III were obtained.

In Fig. 4, gel swelling has been plotted against xanthate sulphur. Both modifiers lowered gel swelling. Gel swelling was linearly related with xanthate sulphur content, the rate of increase of gel swelling with increase in sulphur being approximately the same for modified and unmodified viscoses.

The fact that Carbowax gave a lower curve than C.H.A. indicates faster dehydration for the former contrary to the respective effects of these modifiers on regeneration; i.e., Carbowax apparently gives a more permeable membrane than C.H.A. at a given xanthate sulphur level.

In Fig. 5, gel swelling has been plotted against immersion. C.H.A.-modified yarn showed a slower initial change in gel swelling with immersion.

Table IV gives the results of a study of water swelling at several key points in the continuous spinning process for super tire rayon. In this particular continuous spinning process the rayon after coagulation and hot stretching was treated with hot dilute sulphuric acid

Table V. Effects of Added Na_2CS_3 in Normal Viscose with and Without Zinc in the Spinning Bath

Additions of Na_2CS_3 to viscose, % (containing 3% CHA)	*Zinc content in spinning acid, g.p.l.*	*Partially regenerated yarn*			
		D-value	*Gel swelling at 20-in. immersion*	*% Xanthate sulphur 10-in. immersion*	*% Xanthate sulphur 20-in. immersion*
0	80	5.75	3.4	11.0	6.1
0.5		5.25	4.6	11.2	7.4
0	0	2.0	4.3	4.8	2.7
0.5		2.25	3.9	4.6	2.7

Spinbath in each case contained 90 g.p.l. H_2SO_4 and had a specific gravity of 1.260 at 20°C.

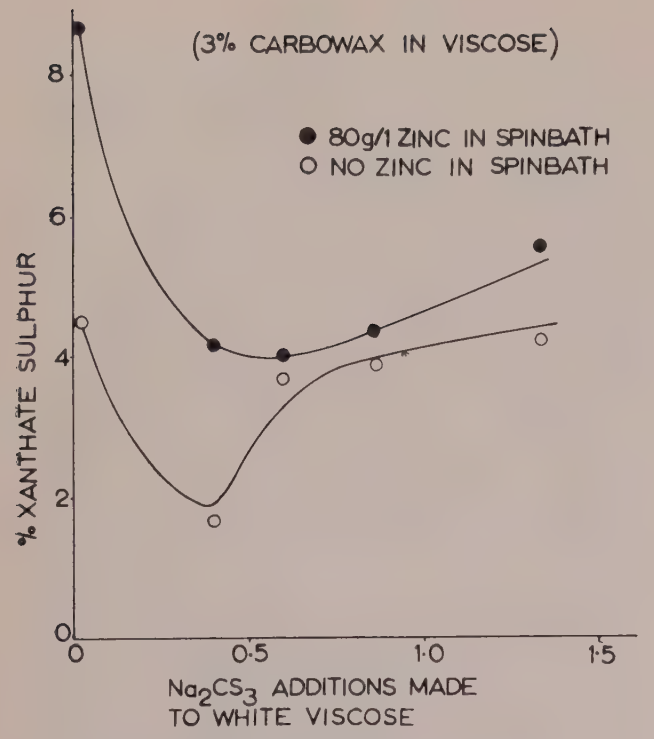

Fig. 10. Effect of trithiocarbonate concentration on rate of xanthate decomposition (20 in. immersion)

(60 g.p.l. at 60°C.) to reduce ash and particularly zinc content. It was then washed with soft water (pH 7.8 at 60°C.) to remove acid and salt, given a lubricating finish, dried to a moisture content of about 5%, and then collected on a ring twister imparting a twist of 1.8 turns per in. to the yarn. The yarn was washed and dried on two pairs of inclined rolls which advanced and spaced the yarn wraps automatically. The machine is illustrated schematically in Fig. 6.

These results again show that modifiers produce lower swelling values than yarn spun from unmodified viscose. This is true at all points in the process. It must be pointed out that swelling of the unmodified yarn decreases at a greater rate than that of modified yarn at each step in the process but the final secondary swelling is still higher than that of the modified yarns.

Effect of Sodium Trithiocarbonate Content in Viscose on Super Tire Yarn Spinning

With the conditions used to prepare super tire yarn viscose in this laboratory, normal Na_2CS_3 content of viscose is 0.60 to 0.75%. In order to investigate the effect of its concentration in viscose, 0.5 and 1.0% was added to two different batches of viscose. The Na_2CS_3 was prepared in advance by reacting Na_2S and CS_2. Yarn xanthate sulphur contents were measured on samples taken just before the first godet (see Fig. 6). Figure 7 shows the results which were obtained.

A second experiment was made in which 0.5% Na_2CS_3 was added to viscose modified with 3% C.H.A. and the viscoses were spun into the regular spinbath and the same bath free from zinc sulphate but with added sodium sulphate to give the same specific gravity. Table V gives the results obtained.

1. In the presence of zinc sulphate, the extra Na_2CS_3 increased gel swelling quite significantly and slowed down regeneration. The latter difference was only apparent at 20-in. immersion. The results of Fig. 6 were on samples taken after 28-in. of bath immersion plus 9 in. of air travel, which show even more marked influence on regeneration, further suggests that the Na_2CS_3 does not exert its retarding effect in the earliest stages of spinning, as was the case with C.H.A. and Carbowax modifiers, but does later on in the process.

2. In the absence of zinc sulphate, extra Na_2CS_3 had little effect on gel swelling or rate of regeneration. This suggests some interaction between zinc and Na_2CS_3.

3. Comparison of the results of the two control batches shows that zinc sulphate had large effects on D-value, gel swelling, and rate of regeneration, in agreement with previous findings (Table I).

In a third experiment, a viscose batch containing 3% Carbowax was purified by anion exchange (*7, 8*) through Amberlite IRA-400. The ion exchange apparatus is shown in Fig. 8. The "white viscose" thus obtained was divided into five parts from which a series containing 0, 0.3, 0.6, 0.9, and 1.3% Na_2CS_3 was prepared by adding the appropriate amounts of this salt. These viscoses were spun into zinc and zinc-free spinbaths. Figures 9 and 10 show the results.

Complete absence of Na_2CS_3 in viscose resulted in abnormally high gel swelling and xanthate sulphur values, at 20-in. immersion. Both gel swelling and xanthate sulphur values decreased to a minimum at Na_2CS_3 concentration of about 0.5% and then slowly increased again with increase in the proportion of Na_2CS_3 in viscose. At all levels of Na_2CS_3, the presence of zinc in the spinning bath raised the level of xanthate sulphur and lowered the level of gel swelling.

A similar experiment as above was attempted using C.H.A. as modifier. However, unlike Carbowax, about 80% of the C.H.A. was removed from viscose by the Amberlite resin, during purification. This was probably due to the conversion of this amount of C.H.A. to cyclohexyldithiocarbamate anion. However the results obtained, despite their inconclusive character, indicated similar trends as obtained with Carbowax.

CONCLUSIONS

Investigations were made on the effects of adding "modifiers" to viscose in the high tenacity rayon spinning process. The two classes of modifiers commonly employed—amines and polyoxyethylene derivatives—were represented by cyclohexylamine and Carbowax 1500. The results of these experiments led to the following conclusions:

1. Viscose modifiers profoundly affected spinning. They greatly reduced the rate of caustic neutralization. Xanthate decomposition was initially delayed, but then speeded up, so that by the time hot stretch was applied, the modified yarns had lower xanthate contents than the unmodified yarns. Water swelling of the yarn was significantly reduced i.e., dehydration was accelerated. For a given modifier, gel swelling was found to be linearly related to the xanthate content of the yarn.

2. Increased amounts of by-product sodium trithiocarbonate in the viscose delayed rate of xanthate decomposition in the later stages of spinning and increased gel swelling.

3. The above effects due to modifiers were not observed without zinc in the spinbath. Since zinc, even without modifiers, was shown to have profound effects on

xanthate decomposition and gel swelling, it therefore appears that modifiers enhance in some way the action of zinc in the initially formed filaments.

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Operation of the 1957 Experimental Sodium-Base Recovery Plant at Consolidated Water Power and Paper Company

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This plant designed for expansion and completed in 1958 has been operated on acid sulphite and NSSC spent liquor singly and in mixtures to obtain recovery data. The sustained operation described confirms validity of the original flow sheet. Solutions to start up problems are discussed. Hitherto unpublished information is given on the burning of sodium-base liquors, on the first commercial evaporator-crystallizer for separating sodium carbonate and sodium sulphide, on the first United States low temperature recovery air heaters and on the Johnstone sulphur dioxide recovery tower. Data obtained are also significant to kraft recovery.

This paper is to report our findings in bringing the soda ash recovery system on stream in an experimental but commercial sized plant.

The design of this plant and its construction was started in 1957. Peculiar problems of design had to be solved; for the requirement was that the plant should be expandable to accommodate the mill's full production without suspending recovery in the experimental plant. It was, therefore, necessary to first design a full scale plant. The full scale layout was then dissected for nonessentials to the experimental plant. Also double use was considered. Thus pumps for one application were purchased sized for another application in the full scale plant. The thickener for this plant would become a dreg's clarifier for the full scale mill, etc. The entire green liquor system, evaporator-crystallizer, and mixing system were purchased and installed for full scale. The instrumentation was installed on a permanent basis for the enlarged plant. The semichemical liquor making system will handle the mill's requirement.

This equipment and the expendable equipment necessary to complete the experimental recovery plant were housed. This structure constituted one half the floor space planned for the final building. The future addition will enclose mainly the big recovery unit and an electrical precipitator. Foundations were cast for future machinery and steel members were sized to carry the future load requirements.

All unit operations shown in Fig. 1 with the exception of the electrical precipitator were simulated in the experimental plant. They were sized to treat the liquor from at least 20 tons of acid sulphite or 50 tons of NSSC. The experimental program calls for operation on a ratio of these spent liquors that would be expected from mixing the collected liquor of the 130-ton sulphite mill and 100-ton NSSC mill.

As unit processes were completed they were operated by liquor recycling or mill production for operator training during the spring of 1958. The date of start-up was considered June 17 when the furnace was first fired with liquor and all other unit operations had been installed.

LIQUOR TREATING SYSTEM AND EVAPORATORS

This process came about by the observed fact that sulphite liquors even when mixed with other sulphur compounds released most of their sulphur to the furnace gases during the charring operation of the combustion. The resultant smelt would contain approximately 50 mol.% Na_2S. If the sodium sulphide could be separated from the sodium carbonate present, and in some manner be sent back to the furnace, a continuous sustained process was practical.

Two paths were open for development. The sodium sulphide could be added to the thick liquor going to the furnace or means could be found to use the sodium sulphide as a neutralizing agent before evaporation and avoid the use of costly stainless steel evaporators. The second path of development was chosen. The major problem to overcome was the known fact that sodium sulphide added to spent liquor in the amounts available could not be successfully evaporated because 40% of the added sulphide would be volatilized as hydrogen sulphide with the condensates. Bradley and Boyer found that the sulphite and lignosulphonates of the spent liquor could be reacted with the added sodium sulphide under certain conditions to quantitatively form thiosulphate and thus effectively tie up the volatile and corrosive sulphur compounds (*1*).

In order to test this part of the process on a large scale the Nekoosa-Edwards Paper Co. installed two spare kraft evaporators and constructed a small concentrator at the Port Edwards mill. In addition, they installed at this location a porous plate diffuser plate-type oxi-

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dizer similar to one which was at that time being successfully experimented with in the kraft industry for kraft black liquor oxidation. Later, when it was found necessary they also simulated a thickener from a tank which was available at this location.

A blend of green liquor and purchased sodium sulphide was mixed with the spent liquor of sodium-base cooks to give a mixture of pH 11.8 which was then settled in the thickener. The overflow mixture was reacted in the air oxidizer and the reacted liquor evaporated in the two-effect LTV to 55% and then concentrated from 55 to 65% in the forced circulation concentrator. The liquor was stored in steam heated tank cars until sufficient was accumulated to burn in the Nekoosa kraft recovery boiler operating as a sulphite recovery unit.

In addition to this work a method of producing pure sodium carbonate from a high sulphidity smelt was developed at the Nekoosa mill. This technique did not utilize an evaporator-crystallizer such as is being used in the present operation. This Nekoosa Edwards work has been described in detail in two articles by Nugent and Boyer (*2, 3*). In addition there have been several articles published describing the theory of the process, the applications and, as well, the research which was conducted up to the time the Consolidated Water Power and Paper Co. 1957 experimental recovery plant went into operation (*4–9*).

Consolidated Water Power and Paper Co. acquired the evaporators and the concentrator which had been used at the Port Edwards mill and installed them in the experimental plant. In addition, an equivalent size thickener, liquor mixing system, and a similar air oxidizer were installed; the major difference being that these were installed on a much more permanent basis and are elaborately instrumented so that the entire system through the evaporators can be said to be automatic.

As a matter of heat economy the second effect evaporator at atmospheric pressure is vented to a surface condenser along with the vapors from the crystallizer. This condenser is operated with the main plant boiler make-up water and thus recovers some 80% of the heat supplied to the recovery plant for evaporation.

Nearly a year's operation has confirmed the reported results obtained at Port Edwards. For this reason we will not further discuss this part of the system in this paper.

FURNACE AND COMBUSTION

Of the many unanswered questions in the process, the most critical was the day by day behavior of the treated process liquor in a commercial recovery unit. The Nepco tests had established that under one set of workable process conditions the sulphur coming into the plant from the spent liquor was released in the furnace, burned to sulphur dioxide, and left the furnace with the flue gases. The sulphide sulphur in the smelt under

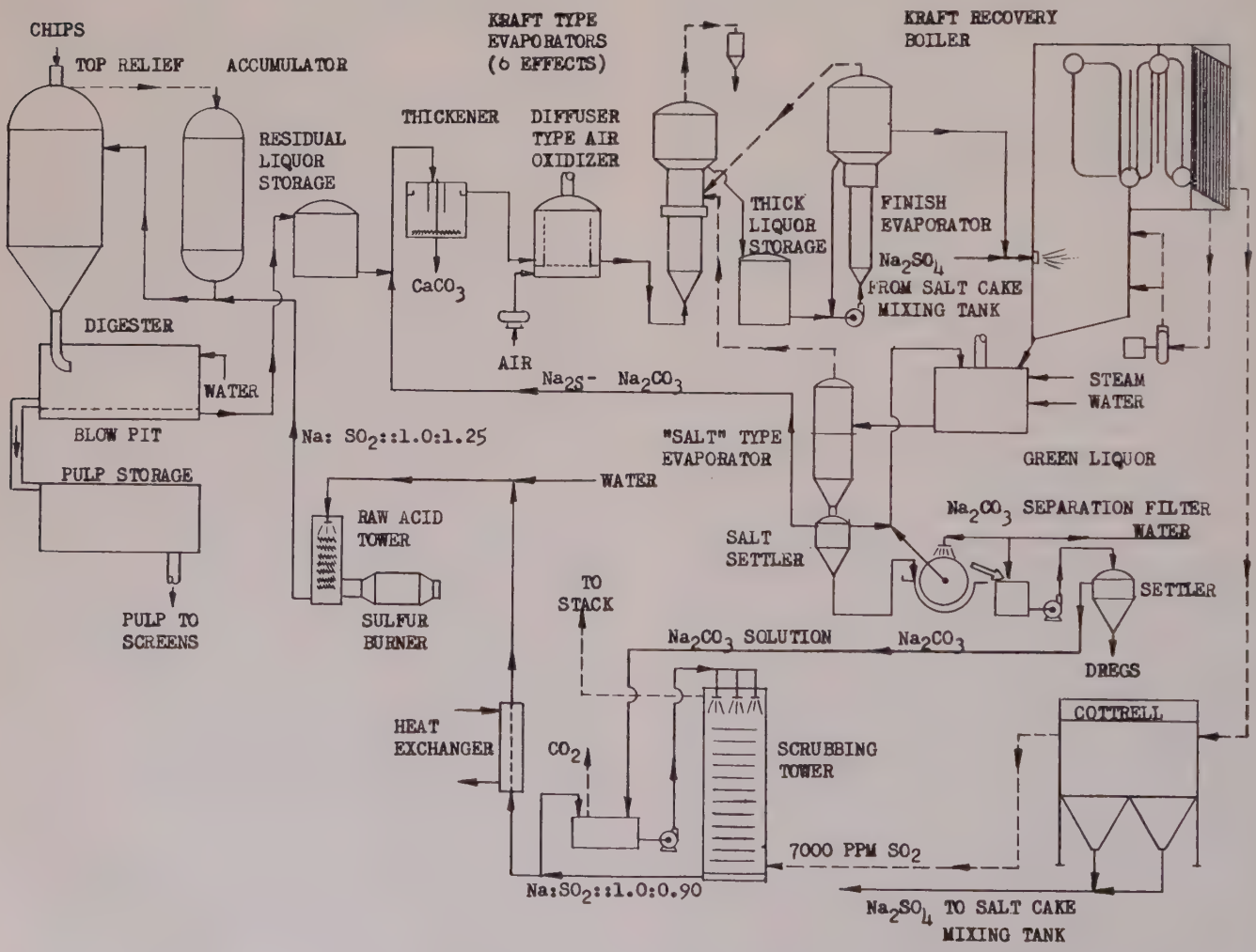

Fig. 1. Complete cyclic process for sodium-base spent liquor acid sulphite pulping

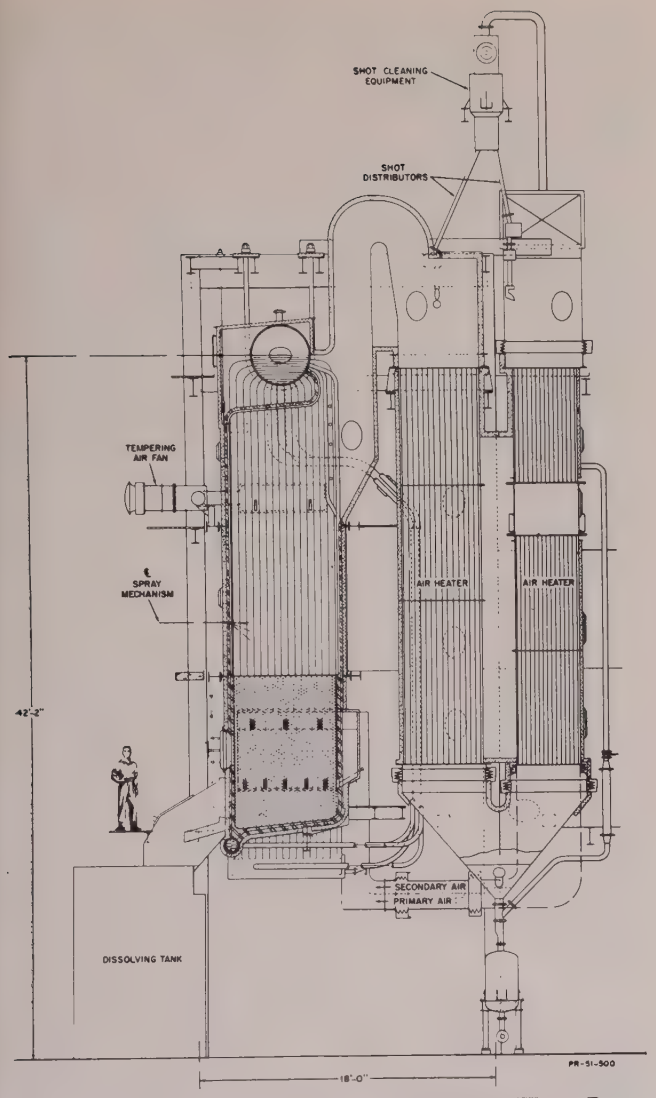

Fig. 2. 20-Ton smelter unit for Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

these conditions acted as a recirculating load throughout the system. This would maintain material balance.

The 1957 experimental plant was fundamentally installed to answer the question of how the sulphur partition in the furnace would act to affect the process under widely varying conditions of feed and combustion. This question was of particular interest to Consolidated in the adoption of the process as their pulp mills would supply varying amounts of acid sulphite and NSSC liquor which had to be recovered and sent back to the respective mills as cooking liquor in the proportions to which the chemicals were received.

The smallest available commercial unit was obtained. It was the B&W smelter rated at 20 kraft tons. Two other smelters had previously been installed and were being employed for the burning of NSSC liquor; a 30-kraft ton unit at Watervliet and a 40-kraft ton unit at Big Island.

Basically, the smelter is identical to the kraft recovery furnace and should have similar burning characteristics to a large recovery unit. The manufacturers assured us that it did have. The similarity, however, ceases at the top of the furnace. There are no superheater and no boiler tubes beyond the furnace outlet. The back furnace wall tubes are so bent to permit an outlet with two sections of screen tubes. For high temperature operation air and water quenching of the flue gases is resorted to. It is necessary that the outlet gas temperature be reduced to at least 1200°F., and preferably 1000°F., so as not to damage the iron duct work or plug the flues with sticky fly ash.

Ordinarily, the manufacturer had supplied the quenching air at about 10 ft. below the top of the furnace. In order to gain a longer burning path we modified the furnace so as to introduce the quenching air at the throat of the outlet flue. The air duct to this point was reconstructed as an envelope surrounding the outlet transition so that the metal temperature of the transition was protected.

This modification removed the confusing factor of the quenching air as an influence upon the furnace operation. It permitted the installation of an oxygen and combustible analyzer at the back of the screen tubes. This could sample the true results of combustion to aid in control of experimentation with the unit. No ash sticking has been observed and the outlet transition did not show signs of burning. What buckling has been observed was present before the modification was made. Altogether this has been a very satisfactory rearrangement.

One serious problem in operating such a small unit was the transportation of the liquor from the concentrator to the furnace spray. The concentrator was situated some 50 ft. away. This problem was solved by pumping a considerably larger flow from the concentrator around the furnace and back into the concentrator thus effectively bringing the concentrator liquor circuit directly to the firing mechanism. A back pressure valve after the take-off point supplies adequate pressure to the firing mechanism. The pipe line to the furnace consists mainly of equipment; that is, a liquor heater, a flow meter, and a flow control valve directly connected to each other and hence into the oscillating spray mechanism. This short equipment line is all that is required to carry the 5 to 10 g.p.m. of 65% liquor going to the furnace.

The furnace is thought to be as completely instrumented as any in the country. The instrumentation was purchased so that it might be used on the full scale unit. Air flow–liquor flow automatic ratioing is employed and provision is made for resetting this ratio with the oxygen analyzer.

The use of a smelter instead of a complete recovery furnace has one less variable which led to some interesting results and required a system modification to duplicate a recovery boiler. A recovery boiler has ash hoppers underneath the boiler and heat traps, and in addition it collects considerable ash in the direct contact evaporator. Most of this collection is of the entrained fly ash or partially burned liquor rather than the volatilized fume which is only collectible in an electrical precipitator or a venturi scrubber.

The extent of this entrainment has not been accurately studied by either manufacturer. As the practical fact in a recovery boiler the entrainment is immediately returned to the boiler in the firing liquor. This ash has a considerably higher sulphur content than the smelt and for this reason has a significant influence upon the sulphidity control. This smelter equipped with air heaters on the other hand passed out all of the entrainment from the system.

When this was realized, study was instituted using a single Multiclone dust collector to follow the entrainment losses. The Multiclone would not be expected to collect any appreciable amount of fume and would, therefore, identify only the entrainment. The Multiclone tests showed that at normal burning rate there is 150 lb. per kraft ton of mechanically collectible entrainment.

We developed the hypothesis that if the collected material were fume it would be a linear function or less of the flows in the furnace, for fume is a partial pressure phenomenon. However, if the collection was of entrainment then the collection should be proportional to the kinetics of the furnace. According to this hypothesis we have established that the collection was entrainment which varies as the square of the burning rate. From the kinetic point of view it must be realized that the ratio of air flow and liquor flow were held constant. Examination of a log-log plot of Multiclone catch versus liquor flow gave a straight line. It showed that the catch C could be expressed as a function of liquor flow L as $C = KL^2$ over the complete range of the furnace capacity.

This experience should aid in explaining to kraft recovery unit operators the limitation of boiler heat absorption which is found on overcrowding a recovery unit. Tube sooting should be a very definite limitation at high firing rates in accordance with our experiments. This is in contrast with electrical precipitation data which would mainly identify fume and shows a linear correlation with firing rate for kraft recovery units.

The salt cake addition was immediately raised to make up for this loss. However, now a 16-tube Multiclone has been installed to prevent the loss and more nearly duplicate the conditions in a boiler equipped unit. It would not be considered a required auxiliary except for an air heater equipped smelter.

The results of the furnace burning, particularly before salt cake addition was employed, caused the revision of our theories used in material balance calculations. The original theory evolved in view of the Nepco tests and all other burning experience had been that no matter what the sulphidity was of the mother liquor added to the incoming spent sulphite liquor that the resulting smelt would be 50% sulphidity, the theory being that the sodium carbonate in the mother liquor would act to hold the sulphur in the smelt. However, with the start of the plant operations on acid sulphite the green liquor was added directly back to the spent liquor until such time as the sulphide inventory would build up and instead the system built up with sodium carbonate. The sulphidity of the smelt got progressively smaller. At this time no fly ash collection or fume collection was being made so that there was no sodium sulphate addition to the furnace. The next attempt to build up the sulphide inventory was to burn straight semichemical liquor. The results were not much better. It was known that the sodium sulphate in the liquor could reasonably go to no other compound than sodium sulphide and that there was an expected fume loss of 150 lb. per hr. of sodium sulphate. For this reason we were justified on an experimental basis of adding to the firing liquor 150 lb. per hr. of sodium sulphate.

At about this time the entrainment losses were realized so that an increase was made in the salt cake addition to compensate for this. The sulphur inventory of the mill increased to where it was possible to extract the process requirement of sodium carbonate with the evaporator-crystallizer and maintain an inventory of sodium sulphide as a circulating load. The 50% sulphidity rule, therefore, appears to be a happy circumstance from the fact that the regular boiler equipped unit which is also equipped with an efficient fume collector returns a considerable amount of highly sulphated soda as ash to the firing liquor. It has been recognized for a long time that the ash and fume sulphation is due to the absorption by the suspended ash of sulphur dioxide.

Further confirmation of our results has been the recent sulphidity results of the burning of a sodium carbonate neutralized acid sulphite liquor at another mill where a 30% sulphidity was observed. We, therefore, concluded that the process control is not automatic; that sulphide inventory is maintained by the addition of salt cake to the system either through ash collectors or the deliberate addition of salt cake. We further conclude from these experiments that the sodium carbonate in the firing liquor does not to any extent capture and retain sulphur in the smelt as was previously thought (*6*).

However, in view of the favorable natural partition there is no risk that without the deliberate excess addition of salt cake the sulphide inventory would ever become uncontrollable. While it has not been tried, at least the theory is in favor of the application of the process to mixtures of kraft with sulphite liquors or the application of the process to two-stage processes where a large yield of carbonate in the smelt would be required.

AIR HEATER

Our work with air heaters in combination with the forced circulation concentrator could have far reaching effect on American recovery practice for the kraft, NSSC, and acid sulphite.

The direct contact evaporator is a heat recovery device. It is a hold over from the original recovery furnace before modern steam generating practice was adopted to sodium base liquor burning. It owes its extended life in the art to the fact that originally the two large boiler manufacturers were unable to adopt the air heater they were using on modern steam boilers to the salt-cake ladened flue gases of their otherwise modern efficient high-pressure steam generating recovery boilers. The introduction of the low capital investment venturi scrubber which also collected salt cake further prolonged the life of the direct contact evaporator.

In the meantime in Scandinavia particularly, where the heat recovery is regarded more highly than in this country, the application of the air heater compatible with modern steam generating practice was aided by the development of the Broman shot cleaning system. It has been reported that there are now over 750 European installations of shot cleaning systems on economizers and air heaters. A considerable number of them are on black liquor recovery units.

The theory of shot cleaning is quite simple. Instead of attempting to blow off the incrustations on the heat traps with air or steam, several tons of steel punchings are dumped through the heat trap gas passes at intervals of, say, 10 min. Random impingement frees the surfaces of even quite adherent incrustations. The soot is

re-entrained in the flue gases and carried out of the unit. The heavy shot are collected at the bottom, blown free of dirt with an elutrating device and then elevated to a storage tank at the top of the unit to await the next cycle. The most favored method for hoisting the shot is with compressed air though other systems are being examined.

The lowest practical outlet temperature of a heat trap is 300°F. The governing factor here is corrosion, at least for the use of mild steel in flue work, induced draft fan, soot collecting devices and stack. The dew point of traces of sulphur trioxide present cannot be reached by the metal temperature without mild steel damage.

The one great advantage of a mild steel direct contact evaporator in the kraft industry is its ability to produce a very concentrated liquor for firing. A properly designed forced circulation concentrator does perform the same function. However, the gases from combustion of the sulphite-type liquors carries a relatively heavy sulphur dioxide concentration which has necessitated acid-proof construction of the direct contact evaporators where they have been used.

In this process we have altered the liquor to be acid precipitable. This precluded the use of a direct contact evaporator as a concentrator, though direct contact evaporator could be used on raw spent liquor before treatment. We were, therefore, obligated to develop the air heater as a final recovery unit heat trap.

We had the knowledge that we would be duplicating European practice and the fact that air heaters with shot cleaning were being operated on black liquor recovery units in this country, however, at temperatures where steam soot blowing could be used. In the face of contrary advice by both the American recovery unit manufacturers, not one, but three air heaters were employed.

The first air heater is simply a gas temperature control device installed on this experimental furnace both for capital investment saving and for flexibility in experimentation. The second air heater is a 12-ft. section. Its characteristics were to duplicate the noncorroding section of the planned full scale air heater. The third section, 8 ft. in length, was to be used experimentally as a corrosion section in order to study the material of construction of the projected full-scale air heater in the cool section wherein it was supposed to corrode. The implication that this section would corrode was based on the unproved factor of whether the low temperature end of the air heater could be kept clean. If it could be kept clean the metal temperature would be high enough to prevent corrosion. The shot cleaning has kept both sections of this heater clean and no corrosion has been observed.

The shot cleaning of all three air heaters' arrangement is shown in Fig. 2. Some incrustations have been observed in the no. 1 air heater. However, this is not serious as the metal temperatures are considerably above the acid dew point. The no. 1 air heater heats waste air which is discharged into the bottom of the stack. The only beneficial effect is to raise the stack temperature and hold down stack corrosion. The waste air is regulated so as to give the proper gas temperature to flue gases entering the no. 2 air heater. The combustion air enters the no. 3 air heater and is passed through the no. 2 air heater to give a 370°F combustion air temperature with an entering gas temperature to 450°F and an outlet gas temperature of the no. 3 air heater of 325°.

With semichemical liquor combustion air temperatures of 500 to 600° can be achieved without difficulty. This additional heat replaces the additional heat which is usually supplied by an auxiliary oil flame to the units operating with direct contact evaporators that are being employed to burn semichemical liquor.

Our operation of these air heaters has led to the following conclusions:

1. Shot cleaned air heaters can be used without difficulty to trap heat to the lowest feasible outlet temperature for recovery unit. The outlet temperatures are comparable to a modern steam generating unit.
2. As the conditions encountered are actually worse than those found with kraft, this conclusion applies to kraft recovery units as well.
3. The judicious use of air heaters can be used to burn NSSC liquor without oil and with a significant saving on capital investment in the required boiler surfaces necessary.
4. Higher combustion air temperatures can also be used to lower the required solids concentration to the unit. The evaporation taking place in the furnace rather than in a direct contact evaporator might eliminate the need for a concentrator, particularly with kraft where dew point considerations do not affect the sulphur recovery as they do with sulphite scrubbing of the stack gases.
5. There are other obvious advantages of the elimination of the direct contact evaporator: (a) the gain of multiple effect economy and electrical energy of the air heater trapped heat by concentrating to full strength in the evaporators rather than single effect economy of the direct contact evaporator. (b) The elimination of the largest single and most uncontrollable source of odor emission, particularly from a kraft unit.

THE EVAPORATOR-CRYSTALLIZER

Historically, probably the first man to recognize the selective solubility of sodium carbonate and sodium sulphide was the great pulp and paper chemist, Viggo Drewsen. Under the direction of Drewsen, Pollock, then with the West Virginia Pulp and Paper Co., developed a temperature composition diagram based on boiling point rise (*10*).

Independently the Brown Co. at the LaTuque mill operated a kraft mill dissolver so as to oversaturate the green liquor. By following the analysis of the mother liquor they established the feasibility of selective solubility as a method of separating sodium sulphide and sodium carbonate (*11*). In the meantime patents were published stating evaporation-crystallization of sodium carbonate sodium sulphide without defining the conditions (*12*, *13*).

In 1951 Western Precipitation Corp. developed the phase diagram of the system Na_2CO_3–Na_2S–H_2O, particularly at high temperatures and of high ratios of sodium sulphide to sodium carbonate, which was the region of interest for evaporation-crystallization. These phase diagrams have been published as part of a patent (*14*). For the past 7 years these diagrams have had wide circulation in the paper industry. At about the

same time The Institute of Paper Chemistry developed the high sodium carbonate end of the diagrams and the lower temperature relationships. These also received wide circulation and are now published as a part of an Institute patent (*15*).

Nugent, Donofrio, and Boyer in 1953 operated a separate dissolver at Nekoosa in which the smelt was blown into an extremely fine slag wool. This established a unit operation in which pure sodium carbonate could be produced by selective solubility in accordance with the phase diagram (*3, 14*).

Parsons, Larsen, and McIntyre of Consolidated Water Power and Paper Co. in 1957 operated a pilot Struthers Wells evaporator-crystallizer installed at the Sulphite Manufacturers Research League Laboratories. The operation confirmed evaporation-crystallization as compatible with the phase diagram. In the meantime the Swenson Evaporator Co. on the basis of the phase diagram supplied to the 1957 experimental plant an evaporator-crystallizer of sufficient size to operate the expanded plant. In August, 1957 McCarthy reported additional laboratory work exploring the high sulphidity end of the phase diagram in which he was seeking the sodium carbonate and sodium sulphide eutectic (*16*).

It was thus that considerable groundwork for the technology had been laid before the first full scale evaporator-crystallizer was placed in operation, but much technological development lay ahead. Many questions were to be answered and could only be adequately answered by the operation of full-scale equipment.

1. Could the system be operated in the presence of the furnace dregs (approximately 2 g. of dregs per liter of saturated green liquor)?
2. Was the production crystal to be the monohydrate (a definite crystal) or the anhydride (a powder)?
3. At what ratio was the sodium sulphide–sodium carbonate eutectic?
4. Would the green liquor sodium sulphate crystallize with the sodium carbonate (as burkite) or stay with the mother liquor?
5. What methods could be used to separate the dregs and sodium carbonate from the mother liquor?
6. Was recrystallization as the hepta or decahydrate (large crystals) necessary for repurification?
7. Would some impurity effect the crystallizer heat transfer surfaces, settler or filtration devices so as to render them inoperable?
8. Could a method of control be developed to produce a uniform product without extraordinary manual attention? This would of necessity encompass the green liquor system as well as the crystallizer.

Much of the attention of the staff has been devoted toward this part of the process and its effect on cooking liquor production. Evaporator-crystallizers are very old and successful unit operations in the chemical engineering arts and yet crystal production is to an extent still an art. Our experience has not been the exception.

Uniformity of feed and consistency of operation are important to crystal production. For this reason, during the start-up period, difficulties that no longer exist effected the crystallizer operation. It was not until October that the plant losses were tightened up so that an inventory of sodium sulphide could be maintained and the crystallizer operated at a uniform rate for crystal production.

In the meantime considerable experimentation in operation had been done using a screen to separate the crystals which were then to be sent to a vacuum filter. The prescreening was abandoned for the following reasons:

1. The dregs would not separate on the screen, but would be returned and built up in the crystallizer system.
2. After an extended period of returning the undersized crystals to the evaporator, the crystal size would deteriorate to where the screen overflow would not maintain material balance.
3. The rotary filter would not pick up the plus 60 mesh without the fine crystals.

The operation to this point led us to the following conclusions:

1. If the dregs were removed at the same rate at which they were brought in, they do not interfere with the crystal size or the operation of the evaporator-crystallizer.
2. At least to 80% sulphidity of the mother liquor the composition or impurities did not effect the crystal size.
3. The steaming rate, particularly during the original crystal formation, did affect the crystal size and the subsequent pattern of crystal production.
4. Certain conditions could exist where the crystals agglomerate and masses of them would float in the salt settler. The cause of this phenomenon is as yet not completely explained, but it is obvious from subsequent observations that entrained air or gas is responsible for the buoyancy.
5. The lighter dregs (mainly carbon) would overflow the salt settler along with the mother liquor. The heavy dregs (mainly iron sulphide) along with a spectrum of crystal sizes could be picked up, filtered and washed on a rotary vacuum filter.
6. The combination of dregs removal in 5 and 7 prevent dirt build up in the crystallizer.
7. Subsequent filtration of the dissolved sodium carbonate crystals solution would remove the iron sulphide dregs. The light dregs were removed from the process when the mother liquor mixed with the spent liquor was settled in a thickener.

The operation of the rotary filter has been subject to considerable alteration from the original plan. The direct filtration of the slurry from the salt settler offers extensive problems:

1. Wire blinding caused either from the dregs or from the precipitated crystal created by flashing.
2. Plugging of the filtrate system due to evaporation of the filter under vacuum.
3. Crystal settling in the filter tank necessitating the development of agitator design.
4. Crystal size control effecting the operation.

The rotary vacuum filter used was rebuilt from a mud washer. Considerable revisions were necessary to suspend the crystals and obtain adequate pick up. The major final conclusions were:

1. The vacuum requirement is 50 cu. ft. of air per minute per square foot of filter surface.
2. The filter media can be a 60-mesh wire over a 14-mesh back-up wire supported by a punched plate drum.
3. Hot water or hot sodium carbonate solution were found necessary for washing as cold water sets up the cake.

4. Wire blinding and filter mechanism caking are prevented with a combination of steam blow back and a steam shower under the doctor knife.

5. Usually during start-up the first crystals are so large they cannot be picked up on the filter and the agitator will jamb. These must be dissolved in the filter tank until a spectrum of crystal sizes are coming to the filter. Then pick up is adequate.

With steam the cake blows free of the wire and slides off the doctor. With proper crystal formation the unwashed cake will run 96.5% Na_2CO_3 and the washed cake will run better than 98.5% Na_2CO_3. Higher purity would demand a repulping and rewashing.

Cooling of the slurry lessens the flashing problems. However, the only way this has been done so far is by adding the secondary filtrate to the filter tank. This put a double load on the filter.

THE SULPHUR DIOXIDE RECOVERY SYSTEM

The absorption system has two separate towers. A large recovery tower treating the furnace gases and a small concentrated sulphur dioxide absorption tower for adjusting the composition of the recovery tower effluent exactly to sodium bisulphite. The over-gas from this tower goes into the flue and is picked up by the recovery tower.

The recovery tower which we have designated as the Johnstone tower is novel for this application. The tower was specifically developed by H. F. Johnstone of the University of Illinois for the absorption of sulphur dioxide from flue gases using a sodium sulphite-bisulphite scrubbing solution. His students over a period of years worked out the entire mathematics of the system (*17*).

It was adopted because of its extremely low pressure drop, low entrainment loss, and simplicity of construction. It approaches the performance of the wetted wall column which is the theoretical standard for absorption. The major problem is the proper material of construction. A very large installation of a similar wood packing was operated on the Fulham Station in London until the war dictated its removal due to the large steam plume. This packing, however, was used with a lime slurry.

The theory of packing wetting was described in a patent issued to Learmonth of Imperial Chemical Industries in which, if certain dimensions are held, the slat packing will completely wet and transfer by surface tension its liquid to the next row of crossed slats without any liquid falling through the gas (*18*).

The density of the tower circulating scrubbing solution is controlled. We have recently installed a titrilog which, it is planned, will control the addition of sodium carbonate to hold the outlet sulphur dioxide concentration to 300 p.p.m. The method which we have used to date is to control the pH at 6.2 of the scrubbing solution entering the tower. The constant volume of the scrubbing solution is controlled by an overflow at the head tank. This overflow becomes the recovered liquor.

The tower effluent is passed through precoated leaf filters which removes the smoke picked up in the tower. The clarified liquor is sent to the auxiliary tower which acts to convert the sulphite-bisulphite mixture to bisulphite. The flow of make up, 18% SO_2 gas from the main plant sulphur burners to this tower is controlled by the pH of the liquor flowing from the tower. This sodium bisulphite liquor is then available to prepare either semichemical liquor by ratioing the addition of sodium carbonate to it or to prepare acid sulphite liquor by absorbing sulphur dioxide in a cooled diluted solution. In the auxiliary tower any sulphide present will be converted to thiosulphate.

The Fulham Station used showers to distribute the scrubbing solution over the top slats which were slanted. As the solution was not economic, entrainment losses were no problem. Johnstone distributed the liquor with small tubes drilled on 1.5-in. centers with a fine hole over each slat and clarified the solution after each pass through the tower. The present tower is equipped with a distributor system that is a result of development of the Chemical Process Dept. of Western Precipitation Corp.

Grooved slanted boards pass the liquid through four levels as spreader plates which feed on to the top row of slanted packing at intervals of 1.5 in. The distributor boards are grooved longitudinally so as to give riffles which will spread the liquid over the boards. Below the riffles on each board are V grooves which guide the liquor on to the next riffled distributor plate. Almost perfect spreading is gained by the liquid flowing over the riffles. As the liquor descends from level to level it encounters double the distributor plates per level. This makes it possible to feed the tower from a few nonclogging large nozzles. Therefore, it is possible to circulate an unclarified solution through the tower.

One of the major problems which we had to solve came from trace materials. The water available for liquor preparation contains small amounts of iron. The recovered sodium carbonate contains small amounts of sulphide which precipitates this iron in an almost nonfilterable minute dispersion. More over, the recovered cooking liquor had to be freed of approximately 6 lb. per hr. of collected soot. The problem was how to filter the mixture. Soot alone on a precoated filter would filter at plant capacity on 20 sq. ft. for 12 hr. without blinding, but in the presence of the ferrous sulphide would only last 1 hr. at the required filtration rate.

It was found that at a pH of 7 the filtration rate was quite impossible. However, if the pH was lowered to 5.8 the filter would act almost entirely on soot. At present, part of the auxiliary tower liquor is added back to the Johnstone tower effluent and the mixture at a pH of 5.8 is filtered. The traces of iron sulphide are solubilized in the process.

Future plans, in which the piping revisions are at present being made, call for the filtration after the auxiliary tower at the pH of bisulphite which is 4.5. Sufficient filtration surface is to be installed to assure 24 hr. service between switching filters.

The filters are 20-sq. ft. vacuum leaf filters with nylon filter media, precoated with approximately 1.5 lb. of Dicalite per 10 sq. ft. The filtrate is extremely clear. After the liquor is converted to bisulphite in the auxiliary tower, the sulphide present is completely destroyed. This eliminates the possibility of again precipitating the iron and discoloring the cooking liquor.

Whether effluent liquor filtration is a necessary part of a full-scale recovery system is not known. It is to be noted that we have no electrical precipitator on the flue gases. Such a dust collector before the tower would be assumed to reduce the collectible carbon by at least

92%. It is reasonable to suppose that with an efficient dust collector the carbon color might be down to the point where it is not necessary to filter the tower effluent. In any event the solution to this problem is to not treat the recovered liquor until the traces of sulphide have been converted to thiosulphate; then it is a simple problem to remove the few smoke particles which remain.

This statement might seem simple like the rest of the problems which have been solved on this plant. However, it has taken many months of operation to establish our following conclusions.

CONCLUSIONS

Liquor Treating System and Evaporators. Sustained operation recycling sodium sulphide gives favorable process conditions for liquor treatment and evaporation. Settling of the spent liquor-mother liquor mixture removes all the particulate material which might interfere in the subsequent oxidizer reactor or evaporators. The exception is pulp which is removed with a side hill saveall. The chemical reaction in the oxidizer is so complete that only a trace of iodine titratable sulphur is found in the evaporation condensates and there is no pungent odor to the condensates. The reacted liquor from the sustained process acts similarily to kraft in standard kraft LTV's to 55% solids outlet concentration. The satisfactory present condition of the first effect mild steel tubes after better than a total of a year's operation confirms that this liquor is at least no more corrosive than kraft. The forced circulation evaporator gives trouble-free operation taking the liquor from 55% to 65 or 70% concentration even when the liquor has added to it the mechanically collected soot plus salt cake equivalent to a precipitator catch.

Furnace and Combustion. Our study with the smelter shows that there is appreciable entrainment from the furnace of a kraft-type recovery unit. The amount is proportional to the square of the firing rate. This function should be a definite limiting factor to the turn up of a recovery unit. Sustained operation confirms that furnacing of the process liquor releases sufficient sulphur and produces sufficient sodium carbonate to sustain the recovery process under widely varying conditions. The previous process theory that excess sodium carbonate added back in the mother liquor would retain sulphur in the smelt is not valid. It is now thought that the additional sodium carbonate over process requirements goes into the smelt unchanged. Salt cake scavenged from the flue gases or added as make up is used to control the inventory of recycled sodium sulphide. The added or returned salt cake goes almost quantitatively to sodium sulphide. The sustained operation has shown that the process cyclic inventory is controllable.

Air Heaters. The shot cleaned air heater can be operated to the lowest feasible temperature (300°F). without trouble. This equipment should be just as effective on a kraft recovery unit. This equipment in combination with the forced circulation concentrator, the effectiveness of which we have demonstrated, should give the kraft industry an economic combination to replace the direct contact evaporator which is one of the large sources of odor emission in that industry. The shot cleaned air heaters on units burning NSSC can be used as heat traps to produce 500 to 700°F. combustion air. This makes it possible to burn NSSC liquor without oil. The combination is particularly effective on a smelter such as we are at present employing.

Evaporator-Crystallizer. The evaporator-crystallizer yields sodium carbonate in accordance with the previously established phase diagram. From inadvertent operation of the equipment the eutectic appears to be 97.5% sulphidity. The furnace dregs do not appear to affect the operation as long as they are removed in the mother liquor overflow and with the sodium carbonate crystals at the same rate at which they are entering from the dissolver. Ordinarily the sodium sulphate present stays in the mother liquor. The combined crystals and heavy dregs are filterable on a 60-mesh screen rotary filter to give a 96.5% pure Na_2CO_3 without washing. Washing can reduce the cake to less than 1.5% Na_2S retention. Greater purity than this would require a rewash. Uniformity of conditions is necessary for satisfactory crystal production. Steam and feed automatic ratioing can be used to produce a predetermined yield of sodium carbonate. Steam blow back and steam washing under the doctor blade of the filter will to a large extent prevent blinding. A proper prefiltration or slurry cooling might aid in the rotary filter operation.

Sulphur Dioxide Recovery System. Further operation of the slat packed tower will be necessary to establish the best materials of construction. Marine plywood does not hold up as well as Douglas-fir. The most satisfactory material under test has been unfinished Consoweld, a phenolic based paper laminate. A simple precoated leaf filter can be used to clarify the tower effluent after it has been reduced in pH to at least 5.8. Traces of ferrous sulphide above this pH markedly interfere with the filtration. Once the traces of ferrous sulphide have been removed they do not reappear on subsequent neutralization to color the liquor.

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Systems Engineering and Operations Research

EDWARD F. THODE

The introduction of a greater amount of scientific rigor to the art of industrial operation and industrial management has been a consistent goal of executives, engineers, and industrial scientists almost since the beginning of the industrial revolution. Systems engineering and operations research are the names given to two related approaches to such a goal. The primary emphasis of the "systems approach" is a rigorous, scientific approach to the "over-all view" of an industrial design or operating problem. The disciplines involved are not new, but the approach, which abandons the steady-state concept, is comparatively new; also, the employment of automatic computer technology as a tool introduces a new element. It may be said that systems engineering is directed at the design and operating problems of production processes and units, while operations research is applied to problems in other areas of management such as sales, marketing, and external finance. As greater scientific rigor is applied to the arts of industrial design and operation, wasteful allowances for human fallibility may be decreased. The conservation of manpower, materials, and money so achieved is the prime motive of systems engineering and operations research effort.

"Comprehensive and systematic evaluation" are the key words describing the interpenetrating arts of systems engineering and operations research. While there are obvious differences between these two regions of scientific effort, the similarities in approach and technique often outweigh the differences. To state that a given problem is within the province of one or another of the disciplines is many times an entirely arbitrary choice. To be sure, there is no difficulty in the classification of a problem on optimum design of a stock-flow and consistency regulator system, on the one hand, and of one concerning the optimum allocation of funds in an institutional investment program, on the other. (It is also obvious that the second is not ordinarily the concern of the technical man.) But, how does one classify the optimization of design and operation of a complete wood pulp manufacturing operation?

Because both of these areas of scientific application warrant the attention of the Technical Association and because the general distinction between the two is not yet clear, it was decided within the Engineering Division that the initial activities of the committees and technical sessions would do well to consider the two jointly.

It is the function of the present paper to define the philosophy and to describe the techniques of the two. Some examples of work in related industries are presented and an attempt is made to delineate areas of distinction.

SYSTEMS ENGINEERING

The writer prefers to describe systems engineering as the comprehensive application of mathematics, engineering science, and cybernetics to problems concerned with the design and operation of large-scale systems and processes. Under this definition, classic examples arise within all the traditional branches of engineering: in civil engineering, water-distribution networks, and automobile traffic control systems; in mechanical engineering, the automatic factory; in electrical engineering, a telephone network such as the no. 5 crossbar dial system; in chemical engineering, an integrated petroleum refinery or a complete alkaline pulping and recovery system; and in industrial engineering, integrated materials handling and warehousing systems. Even though we can cite such examples within specific areas, it is a characteristic of systems engineering that successful prosecution of a project requires the coordinated efforts of a variety of individuals from various disciplines of study, specialists and generalists. Most particularly, the systems approach requires the participation of men of broad training and great breadth of vision, the kind of men often designated today as engineering scientists or scientific generalists.

Thal-Larsen (*1*) has put it succinctly: "It is the recognition of the importance of the 'systems concept' that is new. Often, mistakes have been made in the design of systems. Objectives were not clearly perceived. Properties of the elements that composed the system were not understood. Their interactions were not fully appreciated. Emphasizing the importance of the systems concept has appeared to be one way of minimizing mistakes. This emphasis on the system itself has given us 'systems engineering'—again not new as to practice, but comparatively new as to manner of thinking."

While the effects of systems engineering may be revolutionary (Goode and Machol (*2*) refer to it as bringing about the second industrial revolution, in which man is being replaced by the machine in his control function), the art itself (with its major content of scientific disciplines) is the result of the orderly evolution and progress of science and engineering. Some of the tools are new, some of the concepts represent new syntheses of ideas, but systems engineering is still basically engineering.

OPERATIONS RESEARCH

Now to look at a definition of operations research, such as this one advanced by Churchman, Ackoff, and Arnoff (*3*): "The application of scientific methods, techniques, and tools to problems involving the operations of a system so as to provide those in control of the system with optimum solutions to the problem." In this broad definition, the word "system" usually means an entire industrial organization.

Obviously, the two definitions intersect to an important degree. The "methods, techniques, and

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tools" of the operations researcher are likewise the methods, techniques, and tools of the systems engineer. A breadth of view and training as a scientific generalist is essential to the practitioners of either art. Well-trained operations research men can do very well in many systems engineering problems, and vice versa.

While no one has claimed that systems engineering is a new science, such a claim has been made for operations research. Discussion of this and similar irrelevant points, such as the priority and privileges of "professionals," has generated untoward hindrances to the development of the art. It is to be hoped that such questions may be avoided in the future.

DISTINCTION BETWEEN THE AREAS

As a contribution to the formulation of a distinction between the two areas of effort, the writer proposes the following classification of problems into types: If an industrial problem is primarily concerned with production and internal cost accounting, it belongs in the systems engineering category; if it involves sales, marketing, and external finance to a major degree, then the problem is in the area of operations research. In no case, however, should an important problem flounder because of lack of agreement as to what definition applies.

To aid in the distinction between operations research and systems engineering, we may examine the characteristics of a representative problem of each type. Let us contrast the problem of obtaining optimum quality of white liquor in a neutral sulphite recovery process with that of optimizing the formulation of the dyestuff mixture added to a given grade of colored paper.

Several characteristics of the neutral sulphite recovery problem place it in the realm of systems engineering, namely: (1) the function to be optimized is a well-defined and readily measured material entity or property of matter, (2) the relation of this function to the cost function is not ambiguous (i.e., the optimum cost may readily be ascertained), (3) the system can be effectively isolated from the rest of the enterprise, as far as its cost function is concerned, (4) the various inputs and outputs of the system may be quantitatively described, analytically or statistically.

The dye formulation problem has a much wider scope, as may be seen: (1) Presumably, the function to be optimized is the maintenance of a specific shade on both sides of a production paper; this involves qualities such as hue, saturation, brightness, spectral reflectance, etc., some of which are judged subjectively by the process of color matching. (2) The cost function is not at all clear: What is the dollar value of a color which never varies (perceptibly) in saturation? What value does a customer place on the absence of color two-sidedness? (3) The relation of the cost function to that of the company as a whole is inextricably tied up with basic company policies in areas remote from production: What sort of customers does the company wish to attract? What value does management place on the prestige of having a product which appears never to vary from lot to lot or within lots, even though no particular price premium is attached? How about purchasing policies? Must the ingredients come from different sources because of such policies? What about worker habits and prejudices? Assuming one can specify a formula and the changes in it which should be made as the pulp raw material changes, can the color man, the beater engineer, and the machine tender be persuaded to follow such a plan? Actually, is better control of the pulp raw material needed? And so on. . . . . .

One may readily see that the neutral sulphite recovery problem, even though it involves over 30 variables, can be studied independently. Not all the factors are amenable to complete quantitative evaluation, but they are, at least, subject to estimation with engineering accuracy. The isolatable and primarily technical nature of the problem makes it a systems engineering problem.

The dye problem, on the other hand, cannot be properly studied without consideration of factors extending through the entire range of the company's operation. It involves matters of economics, human relations, and psychophysics more than it does physics and chemistry. The great contribution of operations research is the application of scientific and mathematical techniques to the solution of problems in the former areas. Thus, the dye problem is properly an operations research problem.

NEED FOR SYSTEMS APPROACH

To elaborate on the general question of systems engineering and systems research, let us review briefly the "state of the art" in the process industries:

Until recently, in the field of chemical and process engineering, the condition of "steady state" operation of continuous processes has been universally assumed for convenience in design calculations and in operational control arrangements. Allowance was made for unsteady state conditions (such as involved in a start-up) wherever such conditions are inherent and important, but the transient process upsets and minor input fluctuations found in every real processing system were deliberately ignored in design and control. Oftentimes, if such variations could be accounted for, better quality and yield of product would result. In batch processing also, the best combination of product properties has often been sacrified to convenience of design or of operating conditions. In other words, "safe-side operation" has been the rule, even though excessive materials, energy, and labor may be consumed in the process. The "best" conditions have been too risky from the point of view of continuity of production.

There are two keys to the problem of optimization—the "best" design and operating conditions—of a chemical process or system: One is a sound knowledge of the physical and chemical behavior of the system components and reactants; the other is a quantitative knowledge, or very accurate estimation, of how the behavior of these elements integrates in the actual process. By the use of some of the newer mathematical evaluation techniques and of automatic computing devices, this second key, often very elusive in the past, is now readily available.

PHILOSOPHY AND TECHNIQUES OF THE SYSTEMS APPROACH

In addressing the question of the concept and method of the systems approach, Goode and Machol (*2*)

state: "The concept from the engineering standpoint is the evolution of the engineering scientist, i.e., the scientific generalist who maintains a broad outlook. The method is that of the team approach." They thus summarize in two sentences the earlier part of the present discussion. If, however, the philosophy is that of a broad and coordinated view using a team of individuals with diverse experience and talents, the techniques employed must represent the diversity of the disciplines of the team members.

In addition to the classical engineering disciplines and tools of statics and kinetics, thermodynamics, heat and mass transfer, fluid flow, strength of materials, electronics, and so forth, more unfamiliar names creep in. Take communication and information theory, for example. The larger and more complex a system is, the more crucial becomes the flow of information from one part of the system to another, from machine to machine, from machine to man, from man to machine, and from man to man. Let us illustrate this with the case of a vertical long-tube forced-circulation evaporator, four stories high, installed in a chemical engineering laboratory. This comparatively small and uncomplicated system has 33 valves and adjustments, some dozen indicating gages and meters, and requires two operators, three floors apart. Considering the literal (audible) noise of the steam injector and the instrument "noise" caused by vibrations and pulsations, possibly the greatest obstacle to effective operation is the communications problem.

Probability theory and the related tools of statistical science represent another essential facet of the systems approach. Virtually any large system one can choose to study is affected by random occurrences and inputs. For example, consider the woodyard of a large integrated mill manufacturing hardwood kraft, softwood sulphite, and groundwood pulps. This mill harvests from its own timberlands and purchases from small woodlot owners on an annual contract basis. Arrivals from company operations are subject to a certain amount of control, allowing for the vagaries of the season, but arrivals of contract wood are completely uncontrolled as to amount, species, size, and condition. This mill has a probability problem!

Group dynamics is a relatively new area of study which has a great bearing on systems engineering and operations research. It goes without saying that the well-established science of psychology is badly needed in connection with the design and operation of systems involving human agency, but awareness is only just developing of the importance of a science of group behavior to complement the study of the psychology of the individual. While classical psychology studies the reaction of the individual to his environment and the interaction of individuals in various situations, group dynamics evaluates the response of a *system* made up of individuals to external and internal stimuli.

Cybernetics is the term used to cover a large area of the "new" science exploited in systems work. Cybernetics has often been loosely defined as the science of control, but actually has not been explicitly defined by its founder, Norbert Weiner. Weiner indicates in his pioneering work (*4*) that his principal concern is with two apparent universals of all systems, living and mechanical: One is that all systems appear to be information systems, and the other is that all appear to be servo (feedback) systems. While this is not a definition of cybernetics, it does place the whole question of control and data processing firmly within the purview of this broad discipline. A consideration of this has led Johnson to suggest that all the questions an engineer need consider with regard to a chemical process problem are covered within the three broad domains of thermodynamics, kinetics, and cybernetics (*5*).

Finally there are the tools and approaches involved in a multitude of facets of applied mathematics, including computer technology. In addition to probability theory and statistics, which were discussed earlier, these include the theory of games, numerical analysis, allocation theory, sequential analysis, and, particularly, model theory plus a large class of calculus problems which can be grouped under the heading of mathematics for optimization. In addition to the more familiar items of differential and integral calculus, differential equations and vector analysis, these approaches make extensive use of complex variable theory, conformal mapping, finite difference equations, integral equations, operational mathematics, matrix theory, Boolean algebra, and Laplace transforms. Twenty years ago, most of these words would not even be in the vocabulary of a self-respecting chemical engineer! Today, if he is to participate in systems work, he needs to know, at least, something about their objectives and nomenclature.

Since the engineer is most often concerned with systems models and optimization, these topics are treated in expanded form in the following section.

SYSTEM MODELS

Throughout recorded history, mathematicians, physical scientists, and engineers have made extensive use of various types of models of actual physical systems for convenience in analysis, manipulation, and engineering design. Any model is but an approximation or representation of the "real" system, and the degree of confidence with which a model may be used in exploring the properties of a system depends entirely upon the degree of faithfulness of the representation of the primary object. The advantages of manipulating a model rather than an "actual" object or process are obvious. These advantages take on massive proportions where the system to be studied is nonexistent (e.g., a totally new device or process), is unattainable (astronomical systems), or too large and costly to alter at will (an integrated pulp and paper mill). The primary function of a scientific model is explanatory; it is intended to facilitate determination of how changes in one or more possible aspects of the modeled entity may affect other aspects or the whole (*3*).

It is convenient to distinguish three types of model: iconic, analog, and symbolic. Churchman, *et al.*, (*3*) define these classes as follows:

> "1. An iconic model pictorially or visually represents certain aspects of a system (as does a photograph or a model airplane). 2. An analog model employs one set of properties to represent some other set of properties which the system being studied possesses (e.g., for certain purposes the flow of water through pipes may be taken as the analog of the 'flow' of electricity in wires). 3. A symbolic model is one which

employs symbols to *designate* properties of the system under study (by means of a mathematic equation or set of such equations)."

In chemical engineering work, we can conveniently identify the three types of models: a pilot plant is an iconic model; in such a plant, the effect of operating conditions on, say, reaction rate may be studied. A graph is an analog model; reaction rate behavior may be shown on the graph. Finally, a differential rate equation is a symbolic model which generalizes the kinetic behavior of the system. Note that as one moves from the iconic toward the symbolic model, the flexibility of the model and the ability to generalize with it increases. Altering a pilot plant for the study of variables other than those for which it was originally designed is an awkward and expensive task; even the obtaining of data on variations for which it was designed is time-consuming. Graphical or other analogies may be much more conveniently rearranged than a pilot plant, whereas the manipulation of symbolic models is limited only by the mathematical sophistication and physical knowledge of the analyst. It should also be noted that any model differs from that which it represents in that it does not have all the properties of what is represented. The attempt is made to represent primarily those properties which are needed for the study at hand; this selectivity on the part of the model represents one of the major factors in the economic attractiveness of model experimentation.

Process simulation may thus be recognized as a vital part of engineering practice which has existed since the days of Pythagoras and Archimedes and which came to full flower in the works of the incomparable Leonardo da Vinci. In our day, the perfection of stable electronic amplification devices has given a new twist to the ancient art of study by analogy.

Arnoff has commented about operations research, "OR establishes a model to represent the system under study in order to perform an abstract manipulation on the system (i.e., via the model) and determines how the system can best be changed." (*6*). He has thus very succinctly defined the very core of systems engineering and operations research methodology. Analog and symbolic models are the primary tools of both fields, with the analog model perhaps of greater importance in systems engineering and with the symbolic model—and its handmaiden, the digital computer—predominating in operations research.

Computers

Really, the outstanding "new" feature introduced with the disciplines under discussion is automatic computer technology. Although many problems can be solved by hand methods, it quite often happens in systems engineering and operations research work that the model is too complex to manipulate without recourse to high-speed computation. If the system primarily involves rate processes or other items expressible in the form of differential equations, an electronic analog computer (differential analyzer) can serve as an analog model of the system and be directly operated upon. If the system model is symbolic and involves extensive use of algebra and calculus, and particularly if probability functions enter in, a high-speed digital computer will be required for manipulation of the model.

"Classic" Problems and Models

In the methodology of systems engineering and operations research, there has accrued a number of classic models and problem solutions which may be adapted to a diversity of specific systems with little difficulty. Also, there have been developed certain generalized methods of setting up and manipulating models, which have still wider applicability.

In discussing the question of "Mathematics for Optimization," Amundson (*7*) has summarized the currently employed techniques of applied mathematics used in finding "best values" as follows: calculus of variations, linear programming, dynamic programming, gradient methods, and approximate gradient methods. By and large, these methods assume that the variable to be optimized is related to the independent variables of the system much in the way that the altitude of a given piece of terrain may be described in relation to its geographical coordinates of longitude and latitude. Generalized gradient methods make no limiting assumptions about the contours other than the absence of re-entrant solid angles—"caves" on the "mountainside." Linear programming assumes that all boundaries are straight lines, often a good assumption in the range of interest in process analysis. It is interesting to note that two time-honored empirical approaches to optimization by experiment may be related to the formal mathematical methods. In the gross "one-at-a-time" method of experimentation, one assumes that independently linear relations among the variables control the process. By manipulating but one variable at a time, holding the others constant, it is often possible to find the optimum, exactly, or approximately, depending on the number of variables. On the other hand, if the system is not linear, a number of well-spaced experiments, utilizing the "shotgun" approach, may serve to map out the area of interest well enough to locate the peak representing the optimum. Obviously, here also, the fewer the variables, the more likely the success of such an approach.

In the construction of a linear programming model, it is assumed that the property to be optimized is a linear function of some number of variables. Various other linear relations may exist among these variables, particularly those expressing physical, chemical, and economic limitations on the system. A large number of possible solutions usually exist, and the optimum solution(s) always lies at some limiting values of the variables such as expressed above. Arriving at the best solution becomes a matter of systematically searching the boundaries (limiting values of possible solutions) for the maximum or minimum value of the desired function (*3*, *8*).

In the general case where gradient methods are used, the relations between variables and the limitations on the variables are nonlinear, so that the desired solution is some "peak" (place where some partial derivative of the function vanishes) or rounded contour within the range of interest of the variables. The Box method of experimentation is an example of an approximate gradient method. It is a very ingenious approach (actually a variation of the one-at-a-time concept) to the statistical design of experi-

ments which, by a sequence of experimentation, calculation, experimentation, calculation, etc., permits one to "ascend the peak" with minimum experimentation and manipulation (*8*, *9*).

The formal optimization techniques given above are vital for the solution of large problems but may be out of place with smaller ones. Successful development engineers and scientists have used the "one-at-a-time" and "shotgun" approaches quite effectively for many years and doubtless will continue to use them indefinitely. There is no reason why this should not be; for many practical problems, working out a linear programming approach and its associated computer program would be like limbering up an atomic cannon to blast a brush-covered machine gun nest. The essence of research and engineering art is the selection of proper tools for the job, whether they be old or new, simple or sophisticated.

There are certain classes of problems to which several "standard" models have been applied. These include allocation problems, inventory problems, waiting time problems (queuing theory), replacement problems, and competition (game theory). The recurrence of these problems in operations research has led to the terminology of "operations research tools" or "OR models" in connection with these models and the methods for their manipulation and solution. That is not to say that such approaches are exclusively the province of operations research. Rather, anyone who has a problem which may be related to these areas would do well to examine the standard techniques evolved in operations research (see references *3* and *6*).

APPLICATIONS TO PROCESS INDUSTRIES

Published references to the applications of the systems approach do not contain a great deal of material directly relevant to chemical process industries. For basic material, the interested reader is strongly urged to study all three of the following reference works: "System Engineering" by Goode and Machol (*2*), "Introduction to Operations Research" by Churchman, Ackoff, and Arnoff (*3*), and "Handbook of Automation, Computation and Control" (Grabbe, Ramo, and Wooldridge, editors) (*10*). The original sources, in published papers, are often so condensed and in such relatively obscure notation that a preliminary reading of these general texts is quite essential to learning from the journal articles. The general texts, on the other hand, contain disappointingly few explicit examples drawn from the process industries.

What is virtually a classic paper outlining the systems approach to a chemical process is that of Symonds (*11*) concerning the use of linear programming in determining the balance of petroleum refinery operations. A more recent paper by Eckman and Lefkowitz (*12*) takes the systems approach to the optimization of a chemical batch process, namely, the hydrogenation of vegetable oil. In this instance, a computing-control system was devised and put into operation with an average savings of 23% in batch time over the previous mode of operation. Dealing with the much simpler case of a sintering furnace, Schuerger (*13*) showed that the incorporation of an inexpensive, special-purpose computing device could increase the throughput of a sintering furnace.

The applications of digital computer systems to process control have been discussed by several authors, among them Phister (*14*) and Tierney, *et al.*, (*15*). Recent reviews on analog applications may be found in articles by Williams (*16*), and Russell, *et al.*, (*17*).

A condensed primer for systems studies on a chemical process may be found in a recent paper by Stout (*18*). While this article is primarily addressed toward control system feasibility and design, the method outlined and illustrated applies quite generally to many types of process system studies. Another excellent paper along the same lines is that of James and Boksenbom (*19*). This latter article gives a broad, intensive survey of the mathematical tools for system analysis.

Analog simulation for process control has been an area of systems work which has seen much fruitful activity of late. The paper by Woods (*20*) is a good recent example. In the work reported by this author, the expansion of a scrubber system was simulated in advance to determine the most economical and most readily controllable arrangement of process components. Note that not just the controllers, but the interaction of all units in the process had to be considered and simulated. Among others, Arthur Rose, T. J. Williams, and co-workers have published a number of papers on unit operations simulation and system analysis (*16*).

The references to classic applications of "operations research models" are extensively cited in the general texts mentioned previously. Two of these of particular interest in the very important "transportation problem" are the articles by Charnes and Cooper (*21*) and by Henderson and Schlaifer (*22*). Operations research applications in the pulp and paper industry have recently been discussed by Wolfe (*23*) who cites several examples but does not go into the techniques of solution.

A very good example of the systems approach to optimum design and operation of a chemical process is the recent thesis of Carroll (*24*). In this work, the optimization of a complete kraft pulping operation has been carried out by combining all material and energy balances of the operation into an effectiveness (profitability) function and then maximizing this function, with due regard to limiting factors, by an approximate gradient method.

It is evident from the material now being presented formally at technical meetings and somewhat freely discussed in informal conferences that much good material is now being developed, so that published results of systems work in the process industries should emerge to a helpful extent within the next year or two. We shall then have available excellent examples of the value of the scientific approach to the "over-all view."

CONCLUSION

To meet the pressing demands for better utilization and conservation of raw material and fossil fuel, to decrease the capital investment required per annual unit of production and to increase the productivity of the individual in the battle against inflation, it is essential that all units in our production scheme, from an individual piece of equipment up to an entire manu-

facturing organization operate at their very best. The approach and techniques of systems engineering and operations research can make important contributions toward that goal, particularly by freeing many operating control decisions from the limitations of human fallibility.

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The Fatty Acids of Some American Pulpwoods

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Seven pine and two hardwood species were examined. Paper chromatography indicated that the fatty acid components of the pines were comprised primarily of oleic and linoleic acids, and that smaller amounts of linolenic, palmitic, stearic, and lignoceric acids were present. Arachidic and behenic acids were detected in five species of pine. For one sample each of trembling aspen and paper birch, linoleic acid was the predominant fatty acid, and smaller amounts of oleic, linolenic, and the even-numbered saturated acids from palmitic through lignoceric acid were present. The main part of the fatty acids in all species seemed to be present as triglycerides, but some free acids were present, and in some cases part of the fatty acids appeared to be esterified with other alcohols. From 80 to 90% of the total sterols were in a combined form, possibly as fatty acid esters.

THE fatty acid constituents of pulpwoods are a source of valuable by-products in southern kraft pulping; in other pulping processes, they may be a major factor in pitch troubles and loss of absorbency problems. In spite of their importance, relatively little has been published on the fatty acids which occur in American pulpwoods.

Oleic and linoleic acids appear to be the chief fatty acids in the pines (*1*, *2*) and in paper birch pulp (*3*), and traces of linolenic acid were reported in jack pine (*1*). Most investigators have recognized the presence of relatively small amounts of saturated acids, but often the composition of the saturated acids has not been determined. Kurth (*2*) concluded that stearic acid was present in shortleaf and longleaf pines, while lignoceric acid has been identified in aspen bark (*4*).

Several investigators have examined the fatty acids in tall oil, but most have dealt with European tall oils. A review by Browning and Calkin (*5*) indicated that tall oil fatty acids consist chiefly of oleic and linoleic acids with smaller amounts of palmitoleic, linolenic, erucic (or brassidic), ricinoleic, lauric, myristic, palmitic, and lignoceric acids. Stearic acid has been isolated from a distilled tall oil (*6*), and a branched chain saturated acid has been isolated from a New Zealand tall oil (*7*). The acids present in tall oil were probably present in the original pulpwood, but some may have been formed during the pulping process or during distillation of the tall oil.

In summarizing the results of research carried out at Kolningslaboratoriet, Bergström (*8*) states that the fatty acids in European pine are comprised of oleic, linoleic, linolenic, lauric, myristic, palmitic, stearic, arachidic, behenic, and lignoceric acids. The same acids appear to be present in European spruce, *Picea excelsa* (*9*). Analysis of the ether extract of a European birch, *Betula verrucosa*, indicated that the fatty acids in this species consist chiefly of linoleic acid with smaller amounts of oleic, linolenic, myristic, palmitic, stearic, arachidic, and behenic acids (*10*, *11*).

Developments within the last few years in the field of reverse-phase paper chromatography have provided a rather easy means for the tentative identification of the individual acids in a fatty acid mixture such as may be obtained from wood. Procedures for quantitative estimation of the individual acids on the chromatograms have been described, but some of these were not satis-

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factory in our hands. Gas chromatography of the methyl esters appears to be suitable for quantitative estimations, but the equipment was not available for the present study.

WOOD SAMPLES AND EXTRACTION

In any study such as this, sampling is a serious problem. Ideally, each sample should be taken from a tree of known environment, and a rather large number of samples need to be studied to obtain representative results. Most investigators usually mix a relatively few logs to provide an "average" sample, but such a mixture may not be representative. In the present study, individual pulpwood sticks were examined in order to obtain some idea of possible variations, but the number of sticks studied was far too small to permit any exact conclusions. In addition, the procedure for collection failed to provide any case history for most of the samples.

Samples of the southern pines were obtained in May, 1958, through the courtesy of J. D. Cumbie of Union Bag-Camp Paper Corp. and of Bruce Sanborn of West Virginia Pulp and Paper Co. Two sticks of jack pine were obtained in May, 1958, from H. W. Rowe of Nekoosa-Edwards Paper Co. One stick each of trembling aspen and paper birch were obtained in October, 1958, by Roland Kremers and Miss Barbara Reeder from northern Wisconsin. The aspen had been felled on September 15–16, and the birch was obtained from a commercial cutting operation. Efforts were made to obtain essentially unseasoned wood, but except for the aspen sample the exact date of felling was unknown. As soon as they were received at the Institute, the wood samples were barked and then stored in a freezer until needed.

Just prior to extraction, a disk was cut from each stick and the remainder was converted to sawdust. The sawdust from each stick was thoroughly mixed, a portion taken for moisture determination, another portion taken for extraction, and the remainder returned to the freezer for storage. Data for the wood samples are given in Table I. Diameter measurements were made on the disks after air drying, and heartwood was detected with bis-diazotized benzidine (*12*).

The green sawdust was covered with benzene in a Soxhlet extractor, and after standing overnight was extracted for 6 hr. Benzene does not remove as much extractive as does alcohol-benzene, but it does remove essentially all of the fatty acid constituents (*13*). Green sawdust was extracted in order to avoid any possible changes due to drying and because previous experience had indicated that the highest yields of benzene-extractable material were obtained from undried wood.

FREE ACIDS

The benzene solution from the Soxhlet extraction was extracted five times with 1% NaOH solution in a separatory funnel in order to separate the free acids from the neutral materials. In most instances, the total alkaline solution was acidified directly with 10% H_2SO_4, the liberated acids separated, and these in turn separated into a free fatty acid fraction and a resin acid fraction by selective esterification of the fatty acids followed by saponification of the resulting methyl esters. In later work, calcium chloride was added to the 1% NaOH solution to form insoluble calcium soaps of the higher fatty acids and resin acids which were separated from the other acidic materials.* Selective esterification then was used to separate the fatty acids and resin acids in the acid mixture isolated from the insoluble soaps.

Table I. Data for Wood Samples

Species	*Sample no.*	*Annual rings, no.*	*Diameter, cm.*	*Heartwood, vol. %*	*Moisture, %*
		Southern Woods			
Loblolly pine	1	19	20.8	0.5	45.6
Loblolly pine	2	21	20.1	0.4	49.2
Loblolly pine	3	18	15.9	0.2	48.3
Longleaf pine	1	24	17.9	1.1	37.3
Longleaf pine	2	28	17.8	1.3	48.8
Pond pine	1	23	16.9	2.7	53.2
Pond pine	2	85	31.8	24.0	34.1
Shortleaf pine	1	19	16.3	4.6	44.6
Shortleaf pine	2	38	19.2	9.5	41.9
Slash pine	1	21	20.5	0.9	45.1
Slash pine	2	20	16.3	0.8	45.3
Virginia pine	1	21	12.9	7.4	45.1
Virginia pine	2	34	11.9	14.2	45.3
		Northern Woods			
Jack pine	1	65	15.7	41.6	43.4
Jack pine	2	53	11.8	33.2	46.6
Trembling aspen	1	25	11.0	...	45.1
Paper birch	1	25	10.4	...	28.7

Reverse-phase paper chromatography on oil-treated paper (*14*) developed with acetic acid-water 4:1 and with a peracid developer (*15*) indicated that all of the free fatty acid fractions contained oleic, linoleic, linolenic, palmitic, stearic, and lignoceric acids. Arachidic and behenic acids were detected in at least one sample from all species except for pond and shortleaf pines. It seems likely that these two acids were present in all samples, but that in some cases the amounts were too small to detect. The two hardwood samples gave relatively strong tests for all of the even-numbered saturated acids from palmitic through lignoceric acid. Oleic acid was the predominant acid in all of the pines, while linoleic acid was the chief acid in the two hardwoods. All samples contained a relatively large proportion of linoleic acid, but only small proportions of linolenic acid. Palmitic acid appeared to be the predominant saturated acid in all cases.

The free fatty acid fractions from all samples were relatively impure, and thus the actual content of free fatty acids is less than the values reported in Table II. Paper chromatography on untreated paper (*16*) and ultraviolet absorption spectra (*17*) indicated the presence of pinosylvin and pinosylvin monomethyl ether in the free fatty acid fractions from all of the pines. These and other phenols were mostly eliminated by the calcium chloride treatment, and thus the free fatty acid values obtained when this treatment was made are more reliable than the other values.

Paper chromatography and color tests with acetic anhydride-sulphuric acid indicated that the resin acid fractions from all samples contained typical resin acids, but the procedures did not permit detection of the individual acids. The small amount of resin acid fractions obtained from the two hardwood samples were contaminated with other materials which appeared to be

* This procedure was suggested by J. A. Harrocks.

oxidized or hydroxy fatty acids. Thus, the amount of true resin acids in these two samples was very small.

Paper chromatography indicated that the materials which were not precipitated with calcium chloride consisted of a mixture of phenolic materials. As expected, glycerol in the aqueous layers from all of the saponifications. In a few instances quantitative determinations were made for glycerol, and the results are given in Table III. The quantitative method (*18*) gave theoretical results for a sample of tristearin.

Table II. Yields of Fractions, Ovendry Wood Basis

Species	*Sample*	*Free fatty acids, %*	*Rosin acids, %*	*Other free acidic materials, %*	*Combined acids, %*	*Unsaponifiables %*
			Southern Woods			
Loblolly pine	1	0.05[a]	0.63	...	1.05	0.11
Loblolly pine	1	0.07[a]	0.61	...	1.02	0.10
Loblolly pine	2	0.07[a]	0.32	...	1.00	0.09
Loblolly pine	3	0.50[a]	1.99	...	0.94	...
Loblolly pine	3	0.68[a]	1.94	...	0.66	0.24
Longleaf pine	1	0.06[a]	0.57	...	1.14	0.14
Longleaf pine	2	0.17[a]	0.87	...	0.85	0.23
Pond pine	1	0.10[a]	0.43	...	0.99	0.15
Pond pine	2	0.65[a]	1.98	...	0.77	0.49
Pond pine	2	0.75[a]	0.98	0.92	0.64	0.58
Shortleaf pine	1	0.43[a]	1.48	...	1.25	0.31
Shortleaf pine	1	0.73	1.27	0.32	0.87	0.31
Shortleaf pine	2	0.27[a]	0.86	...	1.23	0.22
Slash pine	1	0.07[a]	0.65	...	1.20	0.15
Slash pine	2	0.45[a]	0.62	...	1.08	0.19
Slash pine	2	0.47[a]	0.60	...	1.01	0.28
Virginia pine	1	0.30[a]	1.20	...	1.14	0.19
Virginia pine	2	0.15[a]	1.00	...	0.88	0.16
			Northern Woods			
Jack pine	1	...	...	...	1.64	0.59
Jack pine	1	0.44	1.53	0.89	1.46	0.47
Jack pine	2	0.14	0.60	0.46	0.85	0.23
Trembling aspen	1	0.10	0.04	0.14	1.22	0.34
Paper birch	1	0.02	0.01	0.05	1.08	0.66

[a] These values include some other materials.

the higher fatty acids appeared to be absent in this fraction.

COMBINED ACIDS AND UNSAPONIFIABLES

The original benzene extract after removal of the free acids was evaporated under reduced pressure to give a neutral fraction which was saponified, and the resulting acid fraction and unsaponifiable fractions were isolated. The acids liberated on saponification are referred to as combined acids because they were present in a combined form in the neutral fraction.

In some cases, sterols were estimated gravimetrically by precipitation with digitonin (*19*). Since only free sterols are precipitated by digitonin, analysis on aliquots of both the original benzene extract and the unsaponifiables permitted an estimation of free and combined sterols. These results are given in Table III.

EXPERIMENTAL

Some procedures were used which have not been published or were modifications of published procedures. These are given in this section.

Table III. Glycerol and Sterol Data

Species	*Sample no.*	*Glycerol, %*	*Weight ratio of glycerol to combined acids unit of*	*Free sterol, %*	*Total sterol, %*	*Weight ratio of total sterol to unsaponifiables*
Loblolly pine	3	0.057	0.09	0.008	0.062	0.26
Longleaf pine	2	0.089	0.10	0.006	0.061	0.26
Pond pine	2	0.039	0.05	...	...	...
Shortleaf pine	1	0.086	0.10	0.013	...	...
Slash pine	1	0.099	0.10	...	...	...
Jack pine	1	0.157	0.11	0.014	0.067	0.14
Jack pine	2	0.079	0.09	0.013	0.065	0.28
Trembling aspen	1	0.105	0.09	0.016	0.108	0.32
Paper birch	1	0.084	0.08	0.011	...	...

Paper chromatography indicated that the combined acid fractions were composed of higher fatty acids with little or no contaminants. The fraction from pond pine sample no. 2 was an exception in that it contained pinosylvin monomethyl ether. There was no evidence for resin acids in any of the combined acid fractions. The combined acids seemed to be made up of the same fatty acids as were present in the corresponding free fatty acid fractions.

Paper chromatography indicated the presence of

Selective Esterification

The total free acid fraction or the acids which precipitated with calcium chloride were dissolved in 20 ml. methanol; 1 ml. of methyl sulphuric acid (1 g. conc. H_2SO_4 per 4 g. methanol), was added and the mixture was refluxed for 0.5 hr. (*20*) (the quantity of reagents was increased proportionally if the sample size exceeded 1 g.). The reaction mixture was diluted with 75 ml. of ether, 50 ml. of water were added, and the mixture was made just alkaline to phenolphthalein by addition of

10% NaOH. The two layers were separated, and the upper layer was extracted twice with 25-ml. portions of 1% NaOH, and washed once with water. The combined aqueous layers were acidified with 10% H_2SO_4, and the liberated resin acid fraction extracted with chloroform.

The combined ether layers were evaporated, the resulting residue of methyl esters saponified, and the liberated acids isolated as the free fatty acids fraction.

Calcium Chloride Separation

The combined 1% NaOH extracts of the original benzene extract was treated with 37 ml. of 10% $CaCl_2$. The insoluble residue was centrifuged, washed with water, suspended in water, and acidified with 10% H_2SO_4, and the liberated acids extracted with ether.

The alkaline solution containing the materials not precipitated with calcium chloride was acidified with 10% H_2SO_4, and the liberated acidic materials were extracted with ether.

Reverse-Phase Paper Chromatography

For the detection of the unsaturated fatty acids, approximately 0.005 ml. of a 2% chloroform solution of the acid fraction was spotted on oil-treated (*14*) Whatman no. 1 paper, and developed by descending chromatography with acetic acid-water 4:1 (v./v.) for 15 to 16 hr. at room temperature. The unsaturated acids spots were detected by exposing the chromatogram to iodine vapor (*21*) followed by inspection under ultraviolet light. The unsaturated acids appeared as dark spots.

Two procedures were used for detection of the saturated fatty acids. In one, the acid fraction was dissolved in glacial acetic acid and 1:1 88% formic acid–30% hydrogen peroxide added. After standing overnight at room temperature, chloroform and water were added, and after mixing the lower chloroform layer was separated. The volume of reagents was adjusted according to the amount of acid fraction in proportions: 14 ml. acetic acid, 2 ml. formic acid-hydrogen peroxide, 2 ml. chloroform, and 15 ml. of water per gram of acid fraction. The resulting chloroform solution was spotted directly on oil-treated paper and the spotted papers were heated in an oven at 80 to 90°C. for 5 min. to fuse the sample in the paper. The chromatograms were developed with acetic acid-water 85:15 for 24 hr. at 37°C., and the spots were detected by forming the copper salts of the acids followed by treatment with potassium ferrocyanide as described by Wagner and co-workers (*22*).

In a somewhat simpler procedure (*15*), the acid fraction was dissolved directly in chloroform (2 ml. per g.) and this solution spotted on oil-treated paper. After heating at 80 to 90° for 5 min., the chromatograms were developed with acetic acid–88% formic acid-30% hydrogen peroxide 6:1:1 for 15 to 16 hr. at 37°C. The acid spots were detected by the copper acetate-potassium ferrocyanide procedure, or the air-dry chromatograms were immersed for 15 min. in a 0.1% mercuric acetate solution containing 0.05% acetic acid. Excess mercuric acetate was removed by washing the chromatogram in running tap water for 45 min. After air drying, the spots were detected by spraying lightly with a 0.2% ethanolic solution of *s*-diphenylcarbazide.

Color Tests for Resin Acids

A reagent which had been used for the determination of cholesterol (*23*) was prepared by adding 40 ml. of cold acetic anhydride to a cold mixture of 10 ml. conc. H_2SO_4 and 40 ml. of acetic acid, and then adding 40 ml. of dioxane which had been purified by passage through an alumina column. One volume of the cold reagent was added to two volumes of a chloroform solution of the material to be tested. This reagent gave the following results with 0.01% solutions of known materials: abietic, neoabietic, and palustric acids gave an immediate blue coloration which rapidly changed to yellow; dihydroabietic acid did not form the blue coloration but gave a yellow color; dihydroabietic acid did not form any coloration even after standing for several hours; isodextropimaric acid formed a yellow coloration on standing for some time; a mixture of oleic, linoleic, and linolenic acids did not form any color within 1 hr. but did form a yellow color for a longer time (a solution containing 0.6% of the same mixture gave a stronger yellow color within 1 hr.); a sterol isolated from tall oil gave a green color within 15 to 20 min. which faded on standing.

The resin acid fractions were chromatographed on oil-treated paper developed with acetic acid-water 4:1, and the resin acids were detected by spraying the chromatograms with an acid solution of diazotized *p*-nitroaniline (*24*). This procedure does not separate the individual resin acids, but does distinguish them from the fatty acids. The spray reagent was prepared by adding 0.13 g. of sodium nitrite dissolved in 1 ml. of water to a cold solution of 0.2 g. of *p*-nitroaniline in 5 ml. of glacial acetic acid and 1 ml. of conce. HCl, and then adding 40 ml. of 80% acetic acid.

Qualitative Tests for Glycerol

The aqueous layer from saponification of the neutrals was neutralized with an excess of sodium bicarbonate, and evaporated on the steam bath in a current of air. The residue was extracted twice with 5-ml. portions of 95% ethanol, and the alcohol solution evaporated to dryness. The residue was taken up in 3 ml. of absolute ethanol, the solution filtered, and then spotted on untreated Whatman no. 1 paper. The chromatogram was developed with ethyl acetate-acetic acid-water 9:2:2, and the spots were detected by spraying with alkaline permanganate-periodate (*25*). Known solutions containing 1% glycerol gave an easily discernible spot, but lower concentrations did not give a detectable spot.

Quantitative Determination of Glycerol

Aliquots of the aqueous layer from saponification of the neutrals were analyzed by the colorimetric procedure described by Smullin and co-workers (*18*).

DISCUSSION

The presence of fatty acid glycerides in wood has been postulated for a long time, but the nature of these glycerides is still uncertain. The usual vegetable fats are comprised of triglycerides with only small amounts of free acids and with no mono- or diglycerides (*26*), although the latter may be formed on aging by partial enzymatic hydrolysis. Fats normally consist of mixed rather than simple triglycerides, and thus fats with eight acid components may contain up to 288 individual

triglycerides (*27*). Separation of the individual glycerides is difficult, and to our knowledge has not been attempted for the wood extracts.

Oleic, linoleic, and palmitic acids are often the major acid components of vegetable fats, and linolenic and stearic acids are often present in relatively small amounts. The higher saturated acids, arachidic, behenic, and lignoceric, have been found in only relatively few fats. Gymnosperm seed fats are characterized by a low proportion of saturated acids consisting of palmitic and stearic, and a high proportion of oleic and linoleic acids with often smaller amounts of linolenic acid (*28*).

The fatty acid components of the fat extracted from pinewoods in the present study were similar to those of the gymnosperm seed fats in that they were comprised chiefly of the unsaturated acids, oleic and linoleic, and in that palmitic and stearic were the main saturated acids. In all of the pines investigated, oleic acid appeared to be the predominant acid, while palmitic acid seemed to be the chief saturated acid. However, previous analyses (*1*, *2*) indicated that the glycerides of green jack pine and the total acids in longleaf pine contain more linoleic acid than oleic acid, while oleic acid was predominant in shortleaf pine and in the free acids of jack pine. These differences may well be due to limitations of the methods used. The present results, as well as other investigations (*8–11*), suggest that small amounts of linolenic, arachidic, behenic, and lignoceric acids are commonly present in wood extracts. Lauric and myristic acids have been reported in European pine (*8*), but their presence was uncertain in the present study. There was some indication that saturated acids with more than 24 carbon atoms might be present, but further work would be required to confirm this.

The fatty acid components of the two hardwood samples tested, differed from the pinewoods in that linoleic acid appeared to be the predominant acid, and the proportion of saturated acids appeared to be higher. European birch also was found to contain a rather low proportion of oleic acid (*10*).

Seasoning seems to result in appreciable hydrolysis of the glycerides, at least in the case of some woods (*3*). If this is generally true, as seems reasonable, the proportion of free to total acids would depend on the time between felling and analysis of the wood. In most of the samples used in the present study, less than 30% of the total fatty acids were in the free form. For the duplicate analyses, the second result for free fatty acids was always slightly higher than the first. Although the difference was usually within the experimental error, this may have been due to a partial hydrolysis even though the samples had been stored in a frozen state between analyses.

For the fatty acid components indicated in wood, a ratio of glycerol to combined acids of approximately 0.11 would correspond to triglycerides; smaller values would suggest the presence of other esters of the acids, while larger values would suggest the presence of mono- and/or diglycerides. Thus, the present results as well as other data (*8*, *10*) suggest that mono- and diglycerides are absent, and that some other fatty acid esters may be present.

The amount of total fatty acid in wood is generally believed to be dependent on the season of the year. However, conflicting results have been published, and it seems likely some of the variations may have been due to other variables. In the present study, the few samples tested prevent any general conclusions in regard to the amount of fatty acids present in the different species, but, of the samples tested, shortleaf and slash pines had relatively high amounts of total fatty acids. The results indicate that one sample of jack pine contained 1.9% total fatty acids, and the second sample 1.0%. Previous data (*1*) indicated that jack pine contains roughly 1% fatty acids. These values are higher than expected because it is generally considered that the yield of tall oil from jack pine is too low to make collection of the skimmings worth while.

The vegetable fats normally contain small amounts of materials including sterols which are termed "unsaponifiables," and a part of the sterol exists in a combined form in the original lipid (*29*). The proportion of unsaponifiables is often high in the lipids from sources which contain relatively little fat. The question of combined sterols in vegetable fats seems to have had but little attention, but many investigators have determined both free and combined cholesterol in animal lipids. In the few samples investigated, from 80 to 90% of the total sterol was in a combined form. This suggests that sterol esters of fatty acids often occur in wood, but this point has not been demonstrated. Possibly, fatty acid esters of other higher alcohols may occur also. It has often been stated that the unsaponifiables in pinewood are chiefly sterols, but in the present study less than one third of the unsaponifiables were sterols.

Mutton (*3*) has recently stated that "it seems likely that the hardwoods do not contain the true resin acids of the abietic acid type." Our results indicate that the two samples of hardwood did contain small amounts of true resin acids, but the amounts were very small.

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A Study of Curl in Decorative Laminates

W. E. MORRIS and J. M. VERDI

Problems relating to the warping of high pressure decorative laminates are discussed with particular reference to processing and environmental factors as well as different types of phenolic resins. The differential in shrinkage between the melamine decorative sheet and the phenolic core with moisture changes are shown to be critical. Shrinkage and water absorption data for laminates after long-term humidity conditioning are presented, and optimum processing and handling conditions to minimize warpage are outlined.

High pressure decorative laminates are normally prepared by the bonding, under heat and pressure, of a number of sheets of phenolic resin-impregnated kraft paper and two sheets; a decorative, alpha-cellulose printed sheet and an overlay sheet impregnated with melamine-formaldehyde resin. This process produces a decorative panel with high strength, good water resistance, and a decorative surface which is highly resistant to scratching or staining. Such laminates have found wide use as counter, sink, and tabletops, as well as in furniture and wall surfacing applications.

One of the most persistent problems encountered in the manufacture of these laminates has been that of warp or curl. Warping presents a problem in finishing operations, packaging, and most important, in the application of the laminates to plane surfaces. Control of the known processing variables to keep the warp within acceptable limits is of prime importance. It was the purpose of this work to study the warping mechanism and to determine the contribution of the variables to laminate warp, in order to establish optimum treating and laminating conditions consistent with the preparation of a satisfactory decorative panel.

SCOPE

Initial studies on decorative laminates indicated that the warp or curl could be changed as the laminate was humidified or dried. A laminate which at atmospheric humidity had a substantial warp, could be brought into a flat state and even curled in the reverse direction by humidification. In drying the laminate at temperatures above room temperature, a more severe warp was produced. Weight change determinations on these panels during humidification and drying cycles indicated that this effect was not only reversible with respect to curl and moisture content changes, but that after repeated cycling the laminate showed approximately the same amount of curl for a given amount of moisture exchanged with the surroundings. Figure 1 shows a laboratory-prepared panel after drying and after humidification. The change in warp is apparent.

This work served to illustrate a condition with which the laminators are only too familiar. During the winter months, particularly in the northern States, when plant relative humidities are quite low, warp is a much more serious problem than in the summer during periods of high relative humidity. Another interesting point observed was that the flatter a panel was when removed from the laminating press the less change it experienced during subsequent drying and humidification cycles.

In order for warp to be produced it would be necessary that a differential in shrinkage or expansion exist between the melamine and phenolic components. It was assumed initially that this differential was somehow connected to the moisture content changes taking place in the two components. Therefore, an effort was made to study the dimensional and moisture content changes of the components independently, since a study of this in a decorative laminate would be extremely difficult.

EXPERIMENTAL

In order to study the phenolic component, laminates were prepared from phenolic core stock alone without the usual melamine print and overlay sheets. Panels were prepared for the melamine study composed entirely of alpha-cellulose, melamine-impregnated print sheets. Previous results had indicated that laminates appeared to warp about as much when surfaced with only a print sheet as when prepared with the standard print and overlay sheet combination. Although the study of phenolic core laminates and melamine print sheet laminates is somewhat removed from the conditions encountered in a decorative laminate, it was believed that when laminated separately at conditions normally used for a decorative laminate, enough of their normal characteristics would be retained to allow a study of the warping mechanism.

This work concerned itself primarily with the study of the effect of various treating and laminating variables on the moisture content and dimensional characteristics of phenolic core and melamine print sheet laminates. Treated stock volatile content was the variable of primary interest, since prior experience had indicated a definite relationship between volatile

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Table I. Summary of Data—Varnish Properties, Paper Properties, and Saturator Conditions

	Condition	*"Plaskon" V-202*	*"Plaskon" V-212*	*"Plaskon" V-201*
Varnish Properties	...	64.0	60.5	70.7
Viscosity, cps.	At 25°C.	305	717	520
Stroke cure, sec.	At 150°C.	105	87	105
Specific gravity	25/25°C.	1.153	1.092	1.200
Saturator conditions				
Web speed, f.p.m. (20 ft. over)	...	24	18	24
Roll settings, in.	...	Scraper bars	Scraper bars	Scraper bars
Temperatures				
Solvent zone, °F.	...	295–300	250–260	295–300
Cure zone, °F.	...	295–300	250–260	295–300
Filler	...	11-mil kraft	11-mil kraft	11-mil kraft
Paper properties				
Resin content, %	...	30.0	28.0	9.4
Flow, (as-treated), %	10 min. at 320°F.	8.6	5.8	28.5
Volatiles (as treated), %				
Volatiles (as-pressed)	10 min. at 320°F.	8.5	7.7	9.8
High, % *	10 min. at 320°F.	4.8	4.9	7.4
Low, % **	300°F.—1500 p.s.i.	8.8	1.9	8.9

* After conditioning @ 50% R. H.
** Dried 10 f.p.m. @ 200°F.

content and warp. Other variables studied were core stock paper type, resin content, and laminating pressure.

The test method of primary concern in this work was one that would allow the measurement of linear dimensional changes on the laminates during their exposure at various relative humidities. A rectangular specimen 2.00 by 4.00 in. was chosen for the dimensional change test. Fine lines were cut with a steel scribe which bisected the specimen along both rectangular dimensions. Measurements were taken with a 0 to 4-in. micrometer along these lines to insure that the subsequent measurements would be obtained at the same location along the laminated specimen. Measurements were taken along both dimensions of the specimens. Since the specimens in all cases showed a greater dimensional change in the cross machine direction, the 4.00-in. dimension was cut in the cross machine direction in order that the change in length to be measured was as large as possible. All data reported herein are for dimensional changes in the cross-machine direction. Figure 2 shows an experimental phenolic panel and test specimen, while Fig. 3 shows similar items for melamine. A thorough check on the reproducibility of the micrometer measurements on a given specimen indicated a precision of ±0.0005 in.

Weight measurements were obtained prior to each dimensional measurement, in order to race the effect of moisture gained or lost on dimensions. Weights were obtained on an analytical balance with the normal precision obtained on such equipment. Time required for weighing was such that weight changes during weighing were about 1 to 2 mg.

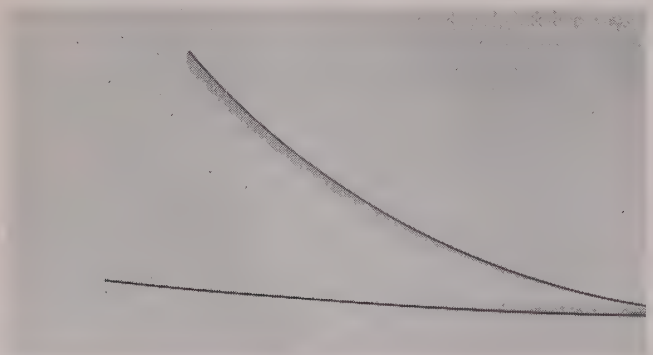

Fig. 1.

Changes in moisture content in specimens were effected by either drying in an air chamber at 50°C. or by humidification in a humidity chamber at a dry bulb temperature of 95°F, and a relative humidity of 85%.

All laminates were prepared at 280 to 290° F. at 1000 lb. per sq. in. for 20 min. at cure temperature. Thermocouples were placed in each panel to trace the cure cycle.

Kraft fillers of 11 mil caliper were used in preparing the phenolic core stock. These fillers were inpregnated with various resins on a horizontal saturator employing a scraper bar arrangement for resin metering. Treated paper properties are shown in Table I. Volatile contents on the phenolic impregnated kraft papers were determined at 320°F. for 10 min.

Melamine print sheets used in this work were purchased as standard stock from a commercial treating concern. The melamine-formaldehyde resin used was identified as a plasticized melamine. Volatile contents were determined at 302°F. for 5 min.

DISCUSSION

Melamine

The data in Fig. 4 were obtained by drying and subsequently humidifying the laminated melamine print sheet panels. The curves show the amount of

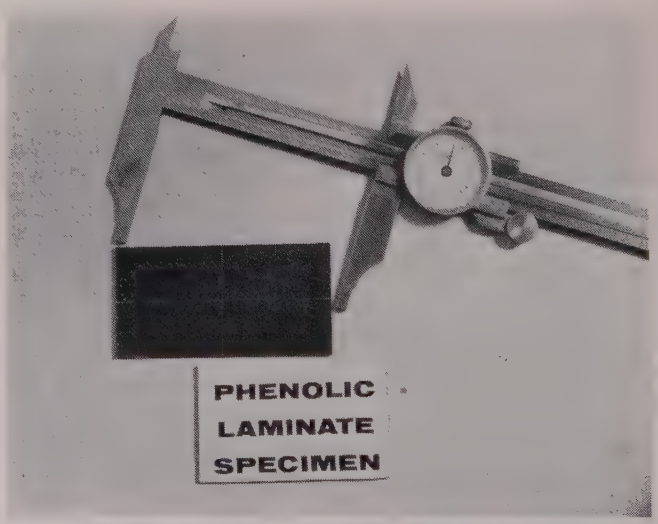

Fig. 2

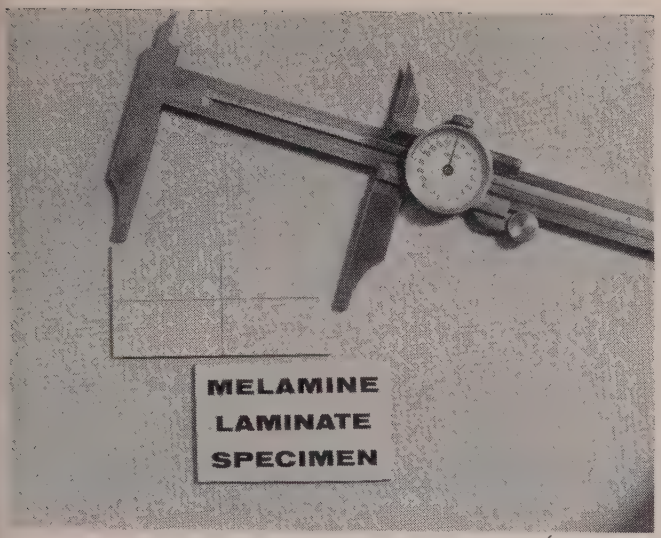

Fig. 3

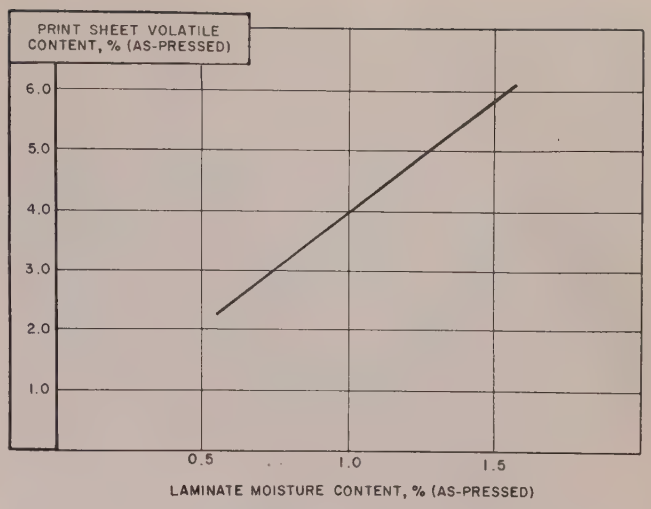

Fig. 5. Effect of print sheet volatile content on as-pressed laminate moisture content

moisture gained by the panels (above moisture-free conditions) and the resultant expansion produced. The data indicate that the "as-pressed" volatile content of melamine print sheets has a direct effect on the equilibrium moisture content of the resultant laminates and thus affects directly the dimensional change experienced by each, due to changes in atmospheric humidity. The curves in Fig. 4 indicate that a considerably more dimensionally stable laminate is produced at a print sheet volatile content of 3.0%.

An interesting point is shown in Fig. 5, where the melamine print sheet volatile content is plotted against the laminate moisture content immediately after lamination. The laminate pressed from 4.9% volatile stock shows approximately twice the moisture percentage as the panel pressed using sheets at 3.0% volatile. Figures 4 and 5 indicate that lamination of print sheet stocks at various volatile contents appears to "build-in" a definite moisture capacity in the laminated section.

In Fig. 12, melamine print sheet laminate expansion per unit weight of moisture absorbed is plotted for a number of print sheet volatile contents. The data fall along a straight line, indicating that a given melamine-paper system has a definite dimensional change coefficient. Lamination at various volatile contents serves only to shift the limits of expansion or contraction to different sections of the curve.

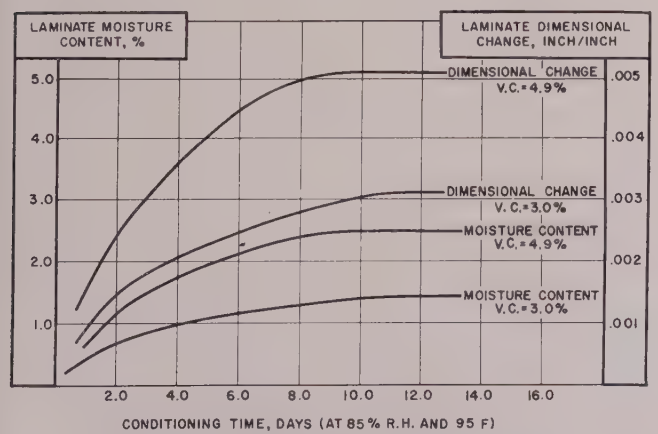

Fig. 4. Melamine print sheet laminates; laminate dimensional change versus moisture content

The absorption of moisture and the resultant expansion in these laminates are approximately reversible, since in a "dry-down" experiment these specimens yield curves essentially indentical to those shown in Fig. 4.

Figure 6 shows the moisture lost by the print sheets during drying in an oven at 91 to 93°C. The print sheets were initially at a volatile content of 4.9%. The sheets very rapidly assume a lower volatile content, with less than 10 min. drying required to assume a volatile content of 3.0%. This print sheet moisture content did not appear to affect the surface appearance of the laminated decorative panels. However, it was found that with the melamine print sheet stocks used in this work, volatile contents below 3.0% produced decorative surfaces with poor flow and gloss.

As indicated previously, dimensional measurements for the melamine panels were taken parallel to the cross machine paper direction. Measurements on these panels in the machine direction indicated a unit dimensional change approximately one-half that in the other direction.

Phenolic

As in the case of the experiment on melamine print sheets, laminates were prepared from phenolic impregnated kraft paper and were subjected to drying (to "moisture-free" conditions) and humidification and the resultant changes in specimen weight and di-

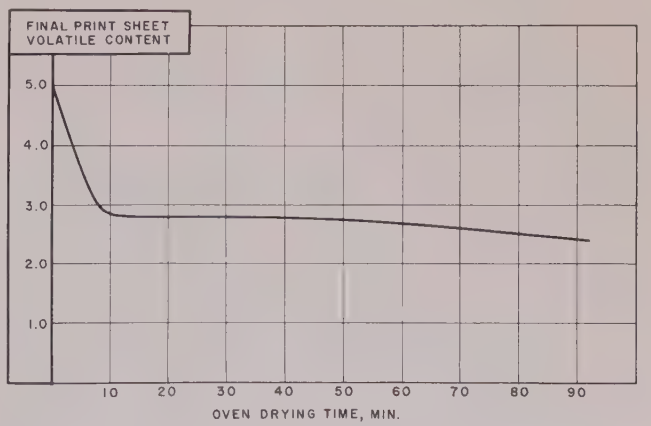

Fig. 6. Oven drying of melamine print sheets at 91–93°C.

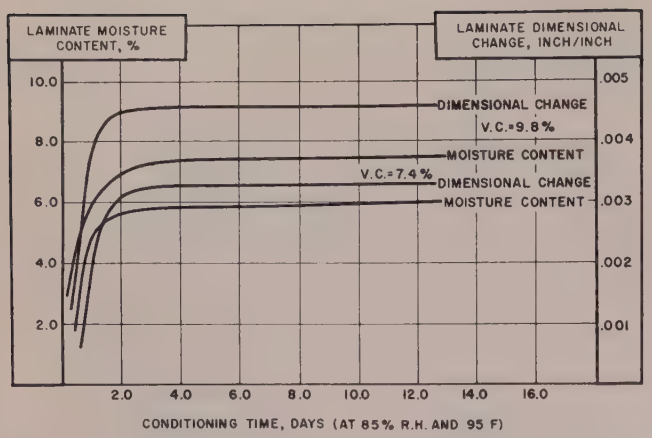

Fig. 7. Phenolic core laminate—Plaskon Laminating Varnish V-201; laminate dimensional change versus moisture content

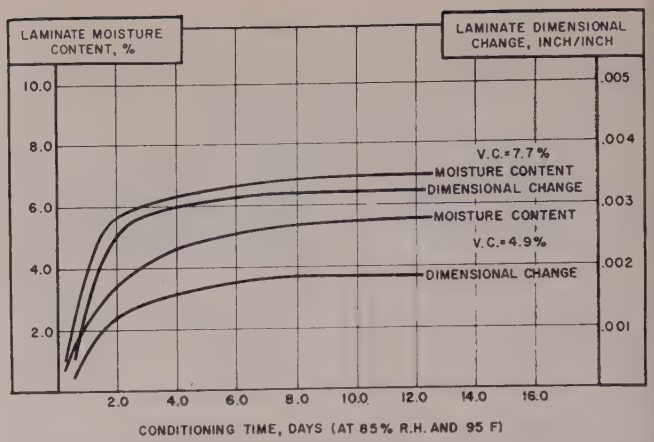

Fig. 9. Phenolic core laminate—Plaskon Laminating Varnish V-212; laminate dimensional change versus moisture content

mensions recorded. However, the additional variable of resins was also considered. Sets of laminates were prepared from each of three different resins. Each resin was laminated at two volatile content levels. Data are shown graphically in Figs. 7, 8 and 9. The three phenolic laminating resins used differed primarily in their paper penetrating characteristics. "Plaskon" Laminating Varnish V-201 is the fastest penetrating resin of the group, while V-212 is the slowest.

Again, as in the case of the melamine specimens, "as-pressed" phenolic core stock volatile content directly affects both the equilibrium moisture content and dimensional stability of the laminated panels. In Fig. 8, for instance, a considerable change in dimensional stability was produced by a change in "as-pressed" volatile content from 4.8 to 8.5%. Similar changes are in evidence in Figs. 7 and 9.

Figure 10 shows a plot of "as-pressed" core stock volatile content against the moisture content of the resultant panels immediately after lamination. It can be seen that the moisture content of the finished panel is proportional to the "as-pressed" volatile content.

In Fig. 12 a curve was plotted representing the phenolic laminate expansion per unit weight of moisture absorbed for all of the panels represented in Figs, 7, 8 and 9. The data fall along a somewhat curved line.

The scatter of the data for the phenolic line was more pronounced than in the melamine data, suggesting that there may also be a small effect present due to resin type. However, there is no doubt that the curve for the phenolic panels differs markedly in slope and location from that shown for melamine. Despite some earlier evidence, an investigation of paper type (northern versus southern kraft), laminating pressure (500 to 1000 p.s.i.) and resin content (30 versus 33%) failed to show any significant effect on laminate moisture and dimensional characteristics due to these variables. If an effect does exist it is of considerably lesser magnitude than that due to changes in core stock volatile content.

As in the case of the melamine panels, phenolic specimens exhibited approximately twice the unit dimensional change in the cross-machine direction as in the machine direction.

Melamine-Phenolic

A plot of total laminate dimensional change against "as-pressed" volatile content is shown for melamine print sheet laminates and phemolic core stock laminates in Fig. 11. The total dimensional change represents the total unit expansion (from "moisture free" conditions) to equilibrium at 85% R. H. and 95°F. Here it may be seen that the melamine panels exhibit a considerably different performance, i.e., equivalent dimensional change at a lower volatile content. Figure 12 shows the dimensional change per per cent moisture

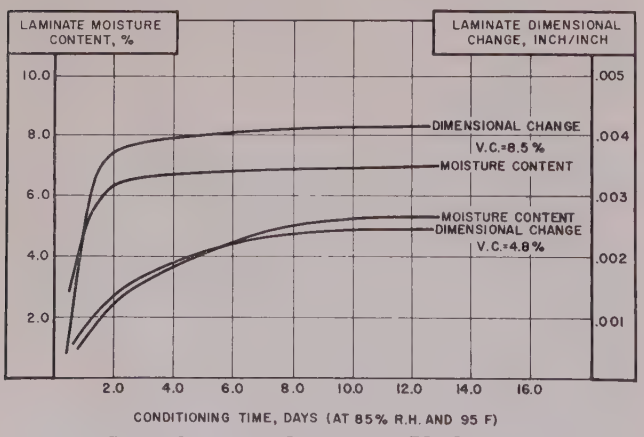

Fig. 8. Phenolic core laminate—Plaskon Laminating Varnish V-202; laminate dimensional change versus moisture content

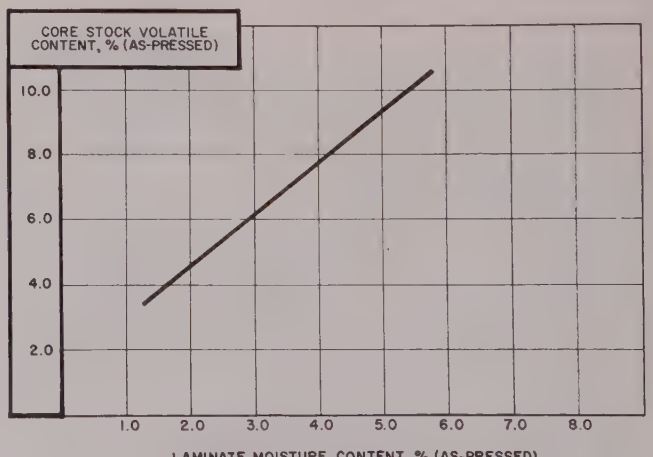

Fig. 10. Effect of core stock volatile content on as-pressed laminate moisture content

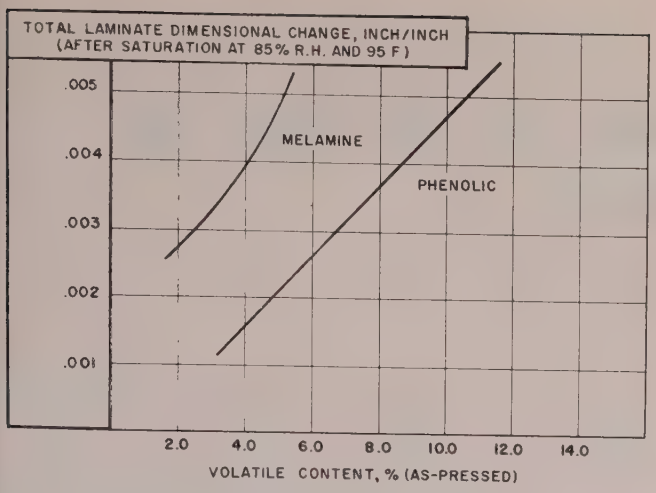

Fig. 11. Total laminate dimensional change as a function of as-pressed volatile contents

gained (or lost) for both materials. The steeper slope of the melamine curve indicates a greater dimensional sensitivity to moisture. However, this is partially offset by the lower equilibrium moisture content for the melamine.

If it is assumed that the conditions for a flat decorative laminate are those wherein the dimensional change for both melamine and phenolic are equal, then Fig. 11 should indicate for both materials approximately those volatile contents which would satisfy this condition. Since this work has indicated a 3.0% print sheet volatile content as a lower limit for good surface appearance, it would appear that the minimum phenolic core stock volatile content necessary for a flat sheet would be approximately 7%. Putting this in terms of a range, a 3 to 4% print sheet volatile would appear to require a phenolic core stock volatile range of 7.0 to 8.5%. Decorative panels prepared in the laboratory using melamine and phenolic stocks at various volatile contents have indicated this recommendation to be reasonably accurate. Furthermore, panels prepared with melamine at 5.0% volatile content and phenolic at 5.0% warped quite badly. Also, Fig. 11 suggests that for melamine print sheets in the range of 4.5 to 5.0% volatile, a phenolic volatile content of 9.5 to 10.5% is required. In practice, such a value for phenolic core would in most instances produce excessive resin flow and bleed-through on the decorative panel during lamination.

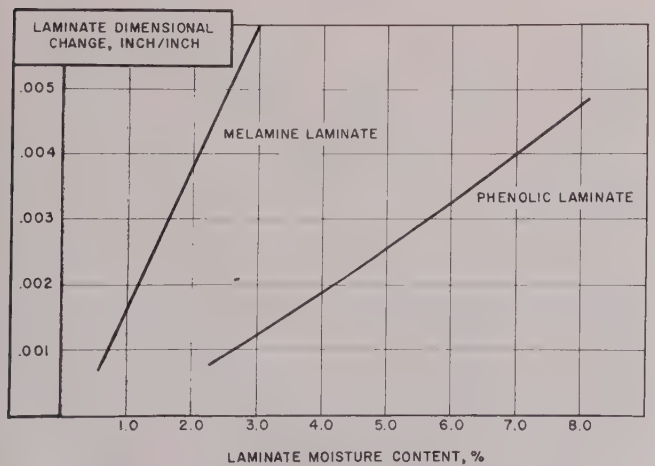

Fig. 12. Dimensional change versus moisture content for melamine and phenolic laminates

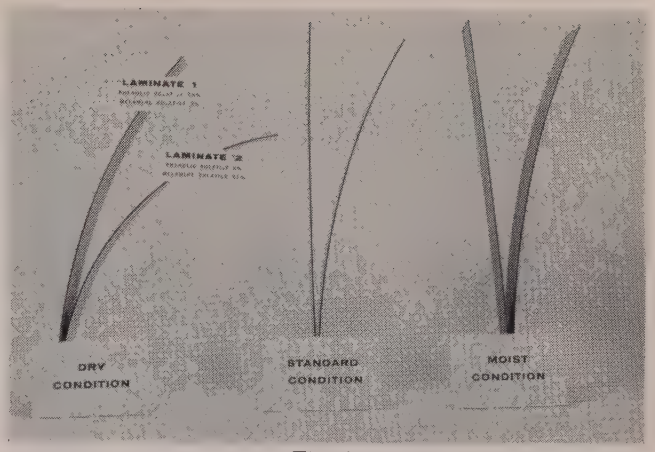

Fig. 13

Most of the data obtained in this work were weight-change and dimensional-change measurements taken on laminated panels during various stages in humidification and drying cycles. These data were taken in an attempt to show that moisture affected the dimensional characteristics of the melamine and phenolic laminated components, and that there were differences existing between the two which caused warpage. However, the warp of a decorative laminate immediately after pressing, as well as its subsequent performance under varying humidities, is fixed during the laminating operation. Dimensional changes take place in the two components during lamination. Although it was not feasible to measure "press shrinkage," it would appear that the volatile content recommendations obtained on the individual laminated components produce the proper dimensional changes during lamination to yield a flat laminate.

The fact that the warp on a freshly laminated panel invariably shows the decorative surface on the concave side, would indicate that the melamine component has shrunk more than the phenolic core. Since the data obtained here seem to indicate that flatness is obtained with the melamine print sheet fairly dry and the phenolic core in a high volatile content condition, press shrinkage would appear to be a function of the moisture content of the stocks prior to lamination. It may be reasoned further that the higher the moisture content of the "as-pressed" stocks, the more shrinkage will be encountered. For a flat panel, therefore, it is desired to have the melamine section shrink as little as possible and the phenolic core as much as possible.

CONCLUSION

Warp in high pressure melamine-phenolic decorative laminates is primarily the result of stresses arising from a differential in shrinkage (or expansion) between the phenolic and melamine components. The "as-pressed" volatile contents of phenolic and melamine impreganted stocks directly affect the warp "as-pressed," as well as subsequent warp due to changing humidities. Flatness is best obtained by holding melamine print sheet volatile contents as low, and phenolic core stock volatile contents as high as is consistent with the preparation of a satisfactory decorative laminate.

Presented at the Thirteenth Plastics-Paper Conference of the Technical Association of the Pulp and Paper Industry, Springfield. Mass.. Oct. 20-22, 1958.

The Stress-Strain Toughness Test for Paper and Paperboard

I. B. SANBORN and R. J. DIAZ

Paper toughness has been defined as the work necessary to rupture paper in tensile load, and a study of the effect which three variables (specimen length, width, and rate of straining) had on stress-strain toughness testing has been carried out. The results obtained indicate that: (1) decreasing specimen width below 15 mm. introduces a negative bias into toughness results, (2) decreasing specimen length introduces a positive bias into toughness results, (3) changes in the rate of straining in the range of from 4 to 100% of the gage length per minute have no statistically significant effect on toughness result. In addition, a round-robin study of a company wide standard toughness test has been carried out on six different instruments, and the results obtained indicate that the accuracy and reproducibility of the test are well within the limits prescribed by the standard method. Finally, the correlation between stress-strain toughness and the bag drop test has been investigated and a good correlation was found to exist between the two tests, indicating that toughness may be a good measure of the "service strength" of bag paper.

ALTHOUGH a considerable amount of information concerning the rheology of paper has been published in the past 15 years, and the general rheological characteristics of it are now understood to at least some extent, there have been few, if any, attempts to devise a standard method of performing stress-strain analyses. The result is that much of the data now available in the literature are not directly comparable because of variations in the techniques by which they were obtained.

During the development of the Clupak process by West Virginia Pulp and Paper Co., it became apparent that a test other than the conventional strength tests was needed if the novel properties of Clupak paper were to be evaluated properly. Stress-strain analysis seemed to offer such a test, and a few exploratory experiments showed that the energy absorbed by paper when subjected to an ever increasing tensile strain appeared to be a good measure of its over-all strength. In West Virginia Pulp and Paper Co. this energy absorbing capacity is referred to as toughness, and is estimated by measuring the area beneath a stress-strain curve obtained by increasing the strain within a specimen of paper at a constant rate until fracture occurs. The purpose of this paper is to present the details of this method of estimating toughness and to outline the result of work aimed at perfecting a satisfactory toughness test and determining if toughness is actually a measure of the service strength of paper.

For purposes of discussion, this paper has been divided into four parts. In the first part, the method of stress-strain analysis developed is described in some detail. In the second part, the results of a study of the effects which variations in specimen length, width, and straining rate have on stress-strain results* are reported. In the third part, the information gathered during a round-robin study of the toughness test on the six stress-strain testers within West Virginia Pulp and Paper Co. is presented. In the last part, brief reference is made to data obtained during a study of the relationship existing between toughness and bag drop results.

A STANDARD METHOD OF STRESS-STRAIN ANALYSIS

One of the first authors to use the term toughness when referring to fiberous structure was H. D. Smith (*1*) in a paper presented before ASTM in 1944. Toughness as used by the technical people within West Virginia Pulp and Paper Co. may be defined as the energy absorbing capacity of paper under tensile load. The term is expressed in inch-pounds of work per 100 sq. in. of paper and is estimated from the area beneath a stress-strain curve obtained by subjecting a specimen of paper to an ever increasing tensile strain until fracture occurs. As a result, stress-strain toughness is a fundamental property of paper just as is tensile strength and stretch.

Even a superficial examination of the paper rheology literature will show that there are a considerable number of testing variables which can affect stress-strain test results. During the early stages of the development of the Clupak process, the pertinent literature was examined and an arbitrary decision was made as to the conditions under which toughness testing was to be performed. The result was that toughness testing was carried out under the following conditions:

1. In an atmosphere conditioned in accordance with TAPPI Standard T402 m-49.
2. At gage length of 5 in., a specimen width of 1 in., and a straining rate of 2 in. per min.
3. Upon a stress-strain tester of good design equipped with either an autographic stress-strain recorder or automatic print-out mechanism.

In addition, it was also recommended that any tester used for toughness testing be equipped with jaws assuring line contact with the test specimen during testing. The data obtained were reported in inch-pounds of work per 100 sq. in. of paper to three significant figures where accuracy permitted, and it was stated that duplicate determinations should not differ

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* The term stress-strain results as used in this paper is a general term used to refer to the tensile strength, stretch, and toughness values obtained upon stress-strain analysis.

by more than 15% of the mean even when carried out on different testers located at different laboratories.

Subsequently, it was learned that jaws assuring line contact with the specimen during testing were essential if accurate stress-strain results were to be

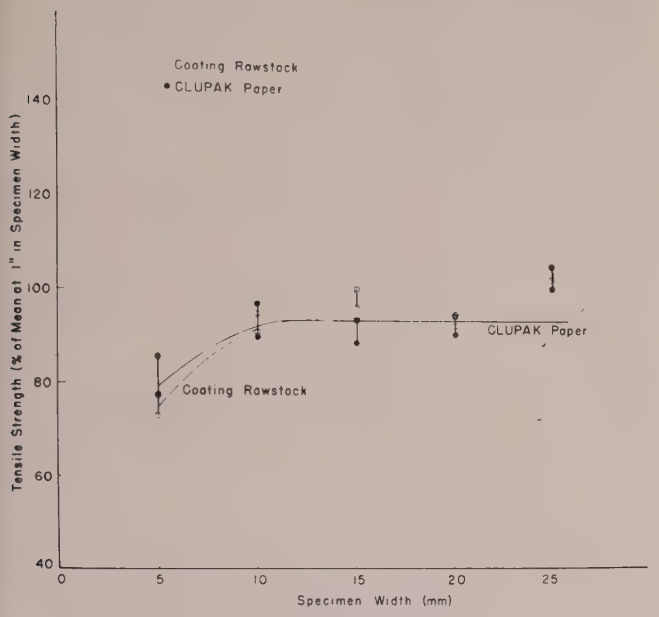

Fig. 1. Specimen width versus tensile strength

obtained and that the reproducibility limits of the toughness determination should be reduced from 15 to 10% of the mean for papers of moderate and high toughness. In addition, it was also noted that errors arising in the recording portion of stress-strain testers could introduce serious biases into toughness results. Consequently, the appropriate changes were made in the method and a copy of it has been attached as an addendum to aid those interested in the details of the test.

A STUDY OF SOME OF THE VARIABLES AFFECTING STRESS-STRAIN ANALYSIS

Because an arbitrary decision had been made as to the gage length, specimen width, and rate of straining to be used during stress-strain testing, a series of experiments were performed to determine if the choice, of testing conditions was appropriate. In general, the results obtained agree with those reported by Setterholm and Kuenzie (*2*), but nevertheless they are presented here in hopes that they will augment the information already available in the literature. The study was carried out with two types of paper, a low stretch coating raw stock and a high stretch Clupak paper. The problem of evaluating the effects of the three variables was attacked by subjecting the two papers to stress-strain analysis on the LRL tester* under the conditions described in Table I.

Table I. Test Conditions

Variable being studied	*No. replicates[a] per determination*	*Specimen width, mm*	*Specimen length, in.*	*Straining rate % Spec., length/min.*
Specimen width	30	5, 10, 15, 20, 25.4	5	40
Specimen length	30	25.4	1, 2, 5, 6, 8	40
Straining rate	30	25.4	5	4, 10, 20, 40, 100

[a] Fifty replicates rather than 30 were used whenever the specimen length was five inches, the specimen width was 1 in., and the straining rate was 40% of gage length/min.

Table II.[a] Effect of Specimen Width Variations

Type of paper	*Sample width*	*Tensile Strength,[b] lb./in.*	*Stretch,[b] %*	*Toughness[b] in.-lb./100 sq. in.*
Rawstock	5.0	12.5 ± 0.4	1.94 ± 0.06	15.1 ± 0.8
Rawstock	10.0	15.4 ± 0.3	2.10 ± 0.06	22.2 ± 0.9
Rawstock	15.0	16.2 ± 0.3	2.14 ± 0.07	22.7 ± 1.1
Rawstock	20.0	15.7 ± 0.3	2.11 ± 0.07	22.1 ± 1.0
Rawstock	25.4	17.0 ± 0.1	2.30 ± 0.03	27.0 ± 0.6
Clupak	5.0	18.9 ± 0.9	12.05 ± 0.31	137.3 ± 8.5
Clupak	10.0	21.6 ± 0.7	12.23 ± 0.26	163.2 ± 6.5
Clupak	15.0	21.1 ± 0.6	12.20 ± 0.35	156.4 ± 5.1
Clupak	20.0	21.7 ± 0.6	12.33 ± 0.22	163.6 ± 5.4
Clupak	25.4	23.8 ± 0.5	12.30 ± 0.20	180.7 ± 4.4

[a] Each determination was made at specimen length of 5 in. and a straining rate of 40% of gage length/min.
[b] The 95% confidence limits of each tensile strength, stretch, and toughness determination as well as the mean are reported.

For purposes of discussion, the presentation of the data obtained has been divided into three parts in which the effects of variations in specimen width, gage length, and rate of straining are discussed separately.

The results obtained during the study of the effect of specimen width variations on stress-strain results are presented tabularly in Table II and graphically

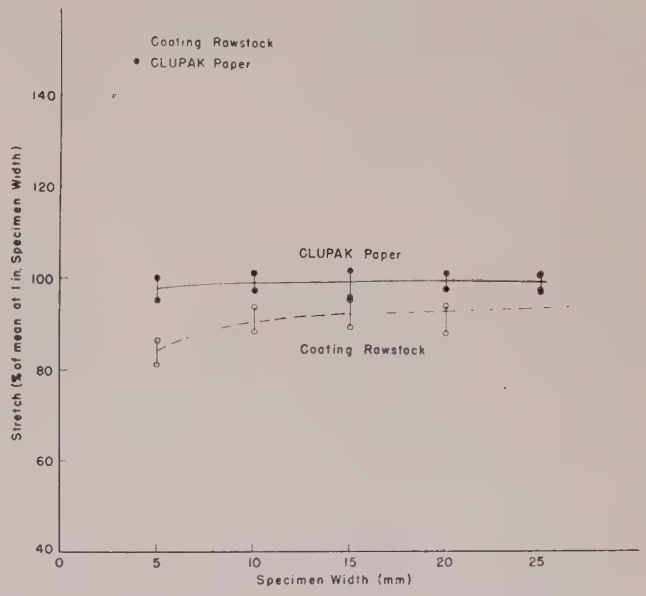

Fig. 2. Specimen width versus stretch

in Figs. 1, 2, and 3.** They show that, although a decrease in specimen width causes a corresponding drop in the apparent tensile strength, stretch, and toughness, the effect is not statistically significant until specimen widths of less than 10 mm. are reached. In addition, it can be seen that an apparent drop in

* The LRL tester is a stress-strain tester designed and built at the Luke Research Laboratory of West Virginia Pulp and Paper Co.
** The 95% confidence limits rather than the means of the data are plotted in Figs. 1 through 6 and 8 through 10.

tensile strength is the primary cause of the reduction in toughness. This behavior is, undoubtedly, caused by the increased influence which edge imperfections have on specimen strength as specimen width is reduced. Thus, it seems that, although decreasing specimen width introduces a negative bias into toughness results, the effect is not serious in the range of specimen widths between 15 mm. and 1 in.

Table III.[a] Effect of Specimen Length Variations

Type of paper	*Sample length, in.*	*Tensile strength,[b] lb./in.*	*Stretch,[b] %*	*Toughness[b] in.-lb./100 sq. in.*
Rawstock	1	18.2 ± 0.2	3.10 ± 0.07	36.3 ± 1.2
Rawstock	2	18.4 ± 0.2	2.68 ± 0.09	33.8 ± 1.2
Rawstock	5	17.0 ± 0.1	2.30 ± 0.03	27.0 ± 0.6
Rawstock	6	18.1 ± 0.2	2.25 ± 0.05	28.8 ± 0.9
Rawstock	8	17.1 ± 0.2	2.12 ± 0.06	26.0 ± 1.0
Clupak	1	27.1 ± 0.6	14.37 ± 0.31	230.8 ± 6.4
Clupak	2	25.8 ± 0.6	13.43 ± 0.30	209.7 ± 6.2
Clupak	5	23.8 ± 0.5	12.30 ± 0.20	180.7 ± 4.4
Clupak	6	23.7 ± 0.5	12.70 ± 0.21	187.3 ± 4.4
Clupak	8	23.9 ± 0.6	12.18 ± 0.25	187.5 ± 5.3

[a] Each determination was carried out at specimen width of 1 in. and a straining rate of 40% of the gage length/min.
[b] The 95% confidence limits of each tensile strength, stretch, and toughness are reported.

Data gathered during the study of the effect which variations in gage length have on stress-strain results are presented tabularly in Table III and graphically in Figs. 4, 5, and 6. From these data it is apparent that reducing specimen length introduces a positive bias into the stress-strain results, but it should be noted that the rapid rise in toughness at low gage length was caused by an even more rapid rise in the apparent stretch. This implied that jaw pull-out might have been responsible for part of the apparent increase in toughness, and the modulus of elasticity of the coating raw stock was estimated from its stress-strain curves as a function of specimen length. The data are presented in Fig. 7 and show that the apparent modulus of elasticity was a function of specimen length. This is proof that jaw pull-out did occur during the experiment, and that the magnitude of the

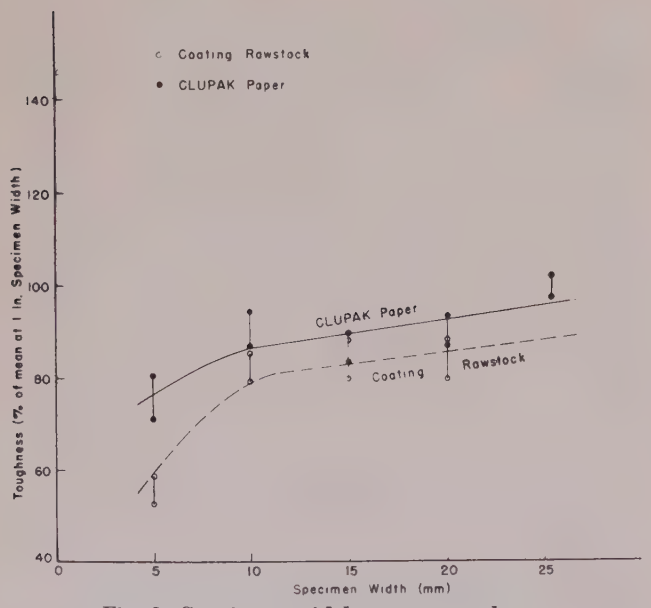

Fig. 3. Specimen width versus toughness

effect which specimen length variations have on toughness and stretch results cannot be estimated from the data available. The data do prove, however, that decreasing specimen length introduces a positive bias into toughness results for the apparent tensile strength is shown to increase significantly as specimen length is decreased. This can be explained by the weak link theory which suggests that the strength of a specimen increases as its size is decreased because there is a statistically smaller chance that a weak spot will be included.

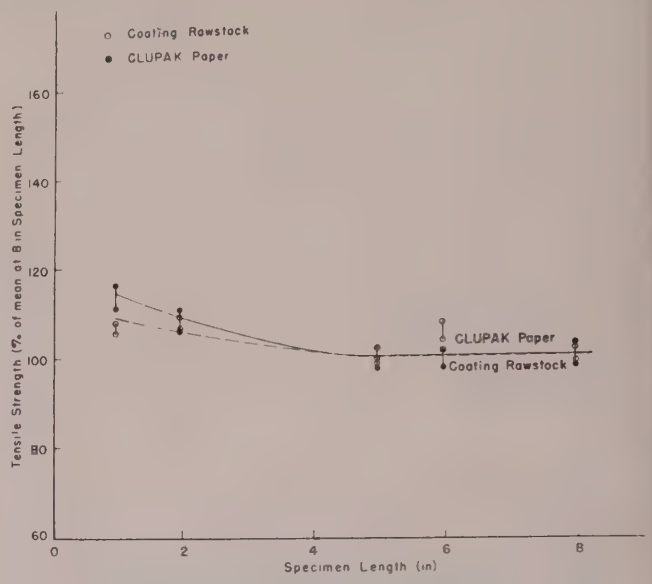

Fig. 4. Specimlh length versus tensile strength

The data presented tabularly in Table IV and graphically in Figs. 8, 9, and 10 were obtained during a study of the effect which changes in the straining rate have on stress-strain results. This information indicates that, although a decrease in the straining rate has a tendency to introduce a negative bias into toughness estimates, the effect is not statistically significant in the range of straining rates studied, despite the fact that the tensile strength of both papers decreased significantly as the straining rate was decreased.

Table IV.[a] Effect of Straining Rate Variations

Type of paper	*Straining rate, % spec. length/ min.*	*Tensile strength,[b] lb./in.*	*Stretch,[b] %*	*Toughness,[b] in.-lb./100 sq. in*
Rawstock	4	14.8 ± 0.2	2.30 ± 0.06	25.1 ± 0.8
Rawstock	10	15.5 ± 0.2	2.29 ± 0.09	25.3 ± 1.4
Rawstock	20	16.1 ± 0.3	2.28 ± 0.07	24.9 ± 1.0
Rawstock	40	17.0 ± 0.1	2.30 ± 0.03	27.0 ± 0.6
Rawstock	100	15.3 ± 0.3	2.11 ± 0.05	20.5 ± 0.77
Clupak	4	20.3 ± 0.5	12.71 ± 0.23	173.2 ± 4.4
Clupak	10	22.6 ± 0.6	13.17 ± 0.23	186.9 ± 5.3
Clupak	20	22.5 ± 0.5	12.67 ± 0.24	161.7 ± 3.4
Clupak	40	23.8 ± 0.5	12.30 ± 0.20	180.7 ± 4.4
Clupak	100	23.5 ± 0.7	12.47 ± 0.22	173.9 ± 6.1

[a] Each determination was carried out at a specimen width of 1 in. and a specimen length of 5 in.
[b] The 95% confidence limits of each tensile strength, stretch, and toughness are reported.

These data also show the effect which poor recorder response can have on stress-strain results, for an examination of the apparent toughness of the low

stretch paper will show that its toughness was significantly lower when tested at a straining rate of 5 in. per min. than when it was tested at lower straining rates. This can be explained if it is remembered that the recorder used with the LRL tester during this series of experiments had a rate of response of about 2 ins. per sec., whereas the low stretch paper when tested at a straining rate as high as 5 in. per min. required a recorder of much more rapid response,

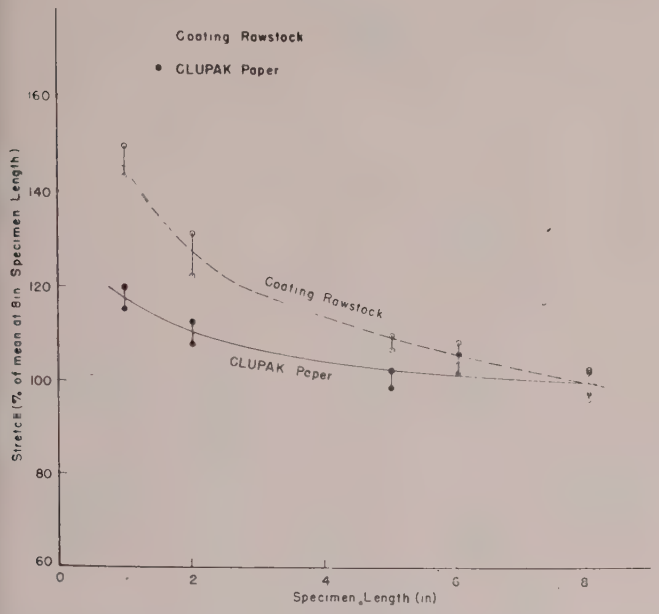

Fig. 5. Specimen length versus stretch

particularly during the early phases of the stress-strain test. The result was that the recorder could not follow the signal sent to it by the strain gage circuit during the test, and a distorted stress-strain curve (see Fig. 11) which gave erroneously low tensile strength and toughness results was recorded.

The conclusions which can be drawn from this set of three experiments are as follows:

1. Decreasing specimen width introduces a negative bias into both toughness and tensile strength determinations.

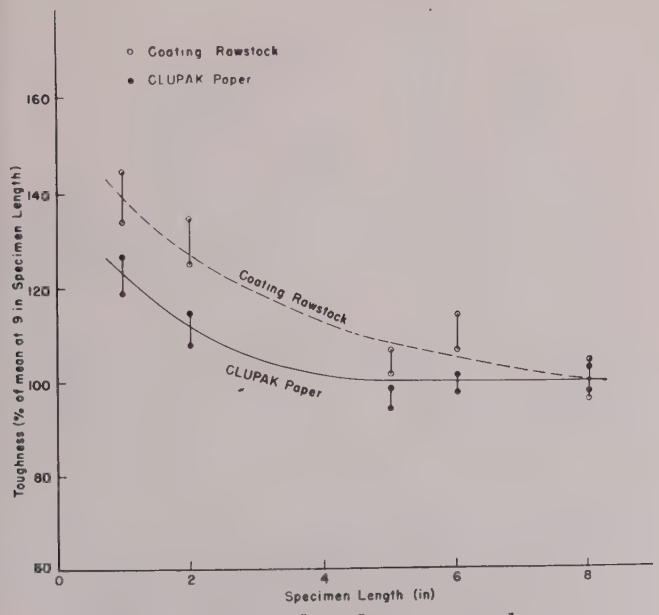

Fig. 6. Specimen length versus toughness

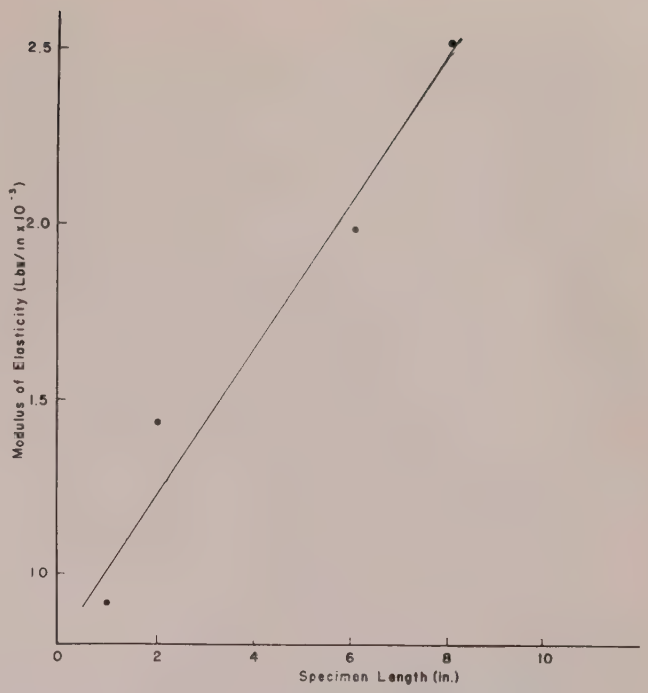

Fig. 7. Specimen length versus modulus of elasticity

2. Decreasing gage length introduces a positive bias into both toughness and tensile strength results.
3. Decreasing the straining rate has no significant effect on toughness results but does produce a slight negative bias in tensile strength determinations.
4. Changes in either specimen width or rate of straining have no significant effect on stretch estimates.

In addition, the data also indicate that the levels of specimen length, width, and rate of straining used by West Virginia Pulp and Paper Co. in its standard stress-strain test do not introduce serious biases into

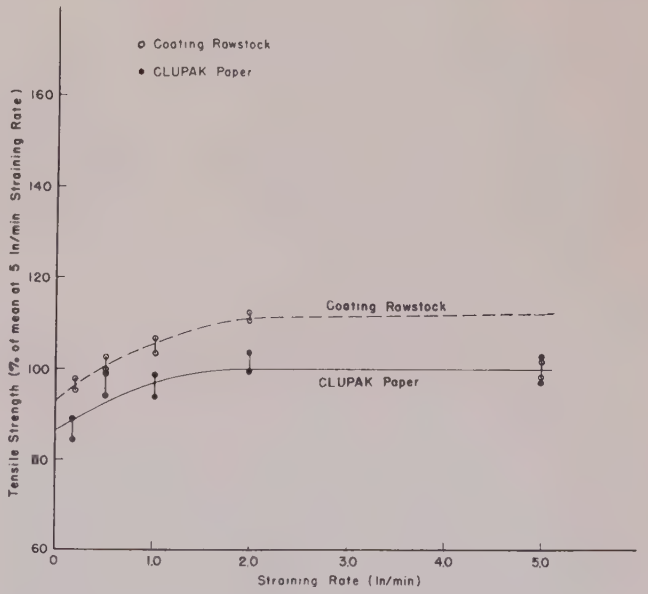

Fig. 8. Straining rate versus tensile strength

the results obtained provided that errors due to jaw pull-out and recorder response are avoided.

A ROUND-ROBIN STUDY OF THE TOUGHNESS TEST

West Virginia Pulp and Paper Co. has six stress-strain testers at its disposal; three table model Instrons,

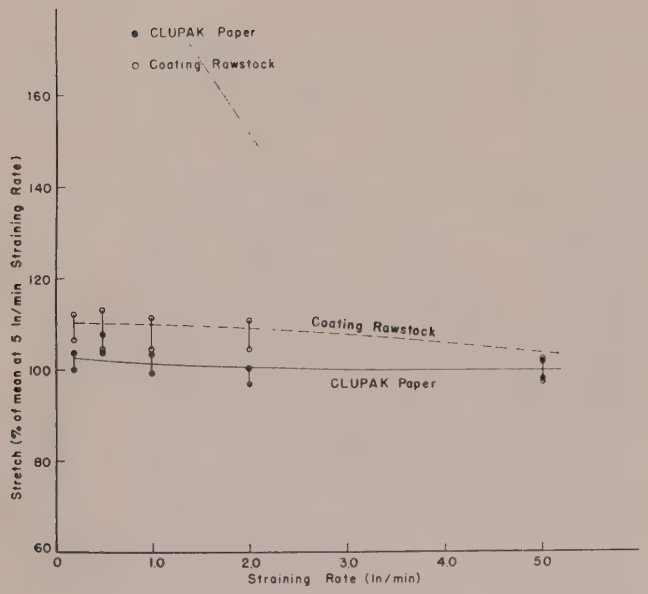

Fig. 9. Straining rate versus stretch

a Thwing-Albert Model 49RC, an STFI and a tester of its own design referred to as the LRL stress-strain tester. These six instruments are located in four different laboratories, and though they are all carefully adjusted to assure their accuracy, they do differ considerably in design. In addition, no two of the

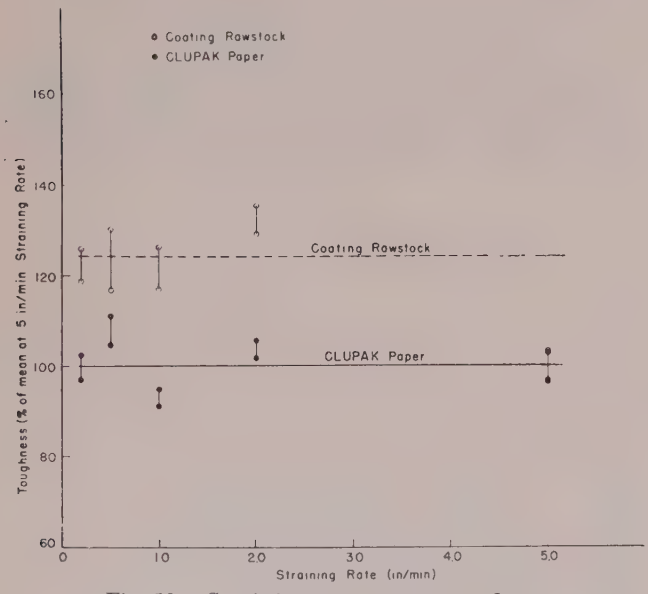

Fig. 10. Straining rate versus toughness

testers were equipped with the same type of jaws at the time when this study was carried out. Therefore, a round-robin study of the company toughness test on the six instruments was performed to see if comparable results could be obtained.

Five types of paper were used in the study, a 35-lb. coating raw stock, a 50-lb. kraft multiwall sack paper, a 50-lb. Clupak kraft multiwall sack paper, a 50-lb. creped kraft multiwall sack paper, and a 32-lb. newsprint. Fifty, 1 in. wide specimens* of each paper were tested on each instrument according to the procedure for stress-strain testing included in the addendum; and the tensile strength, stretch and toughness of each

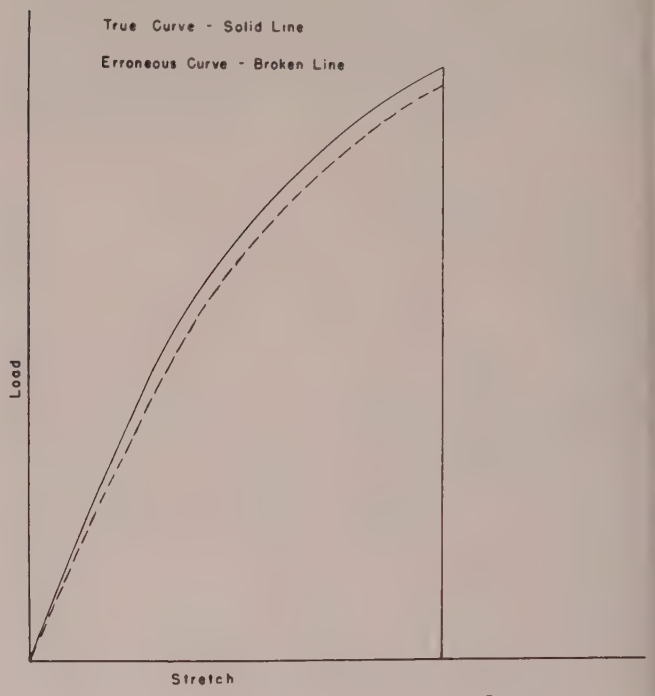

Fig. 11. Load versus stretch

sample tested on each instrument were obtained from either an autographic recorder or an automatic printout mechanism.**

The mean tensile strength, stretch, and toughness of each sample is presented in Figs. 12, 13, and 14

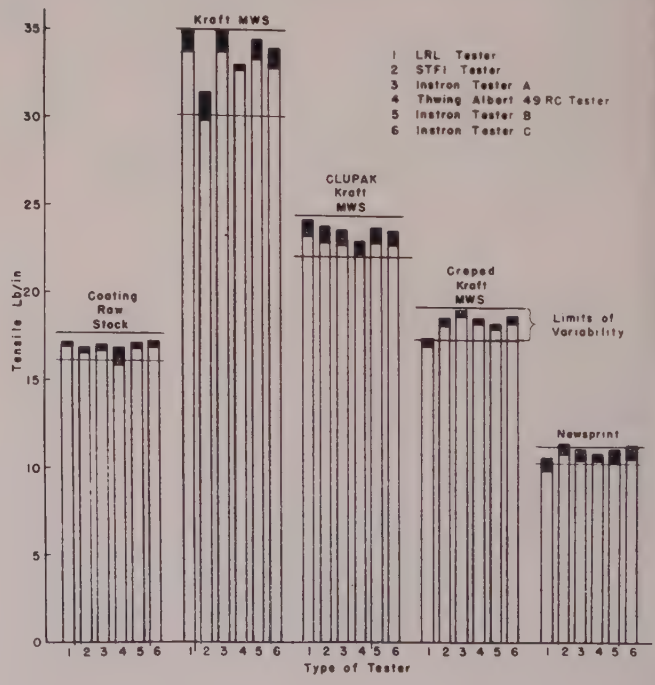

Fig. 12. Type of tester versus tensile strength

along with their 95% confidence limits. These data show that at the 50 replicate level the tensile strength, stretch, and toughness results obtained from the various testers generally fell within the limits of

* Due to physical limitations, specimens tested on the STFI tester had to be tested at a width of 15 mm., a length of 10 cm., and a straining rate of 1.34 mm. per min.

** Only Instron tester C was equipped with an automatic printout mechanism.

variability specified by TAPPI Standard T404 m-53, T457 m-46, and the company standard toughness test. Nevertheless, differences between some of the results at the 95% level of confidence can be seen even at the 50 replicate level. The primary cause of these

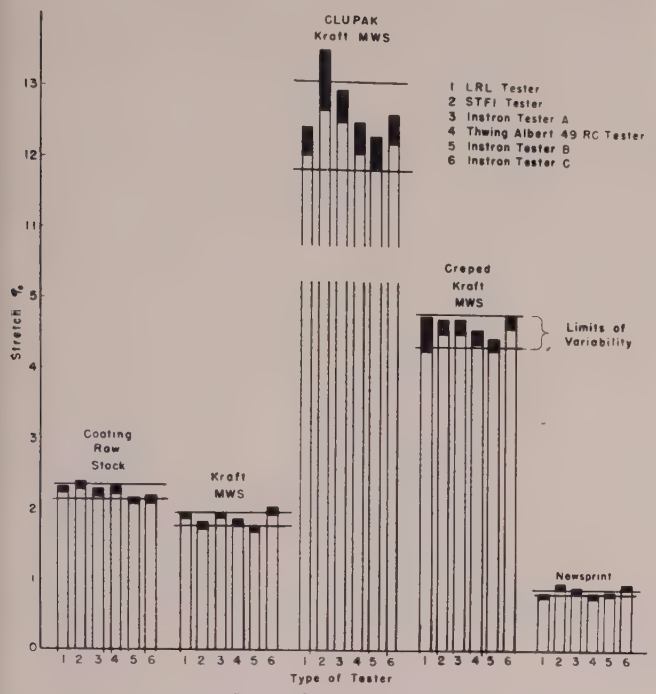

Fig. 13. Type of tester versus stretch

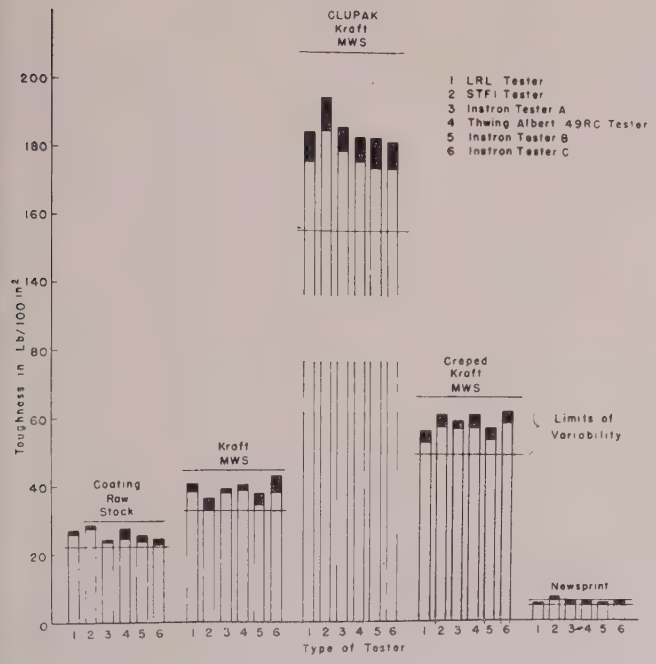

Fig. 14. Type of tester versus toughness

differences is jaw pull-out, and as a result the effectiveness of the various types of jaws in eliminating jaw pull-out can be estimated if it is assumed that all testers were operating properly and calibrated correctly. The jaws used in each instrument are sketched schematically in Fig. 15, and those used by the table model Instron B have been assumed to be most effective in eliminating jaw pull-out, for they maintain line contact with the test specimen throughout a stress-strain test, and either eliminate or reduce jaw pull-out to a bare minimum. Comparison of the results obtained with Instron tester B with those obtained from the other testers† lead to the following conclusions.

1. Differences in jaw design had little or no systematic effect on tensile strength results.*
2. Flat faced jaws such as those used in the STFI and table model Instron A allowed the greatest amount of jaw pull-out

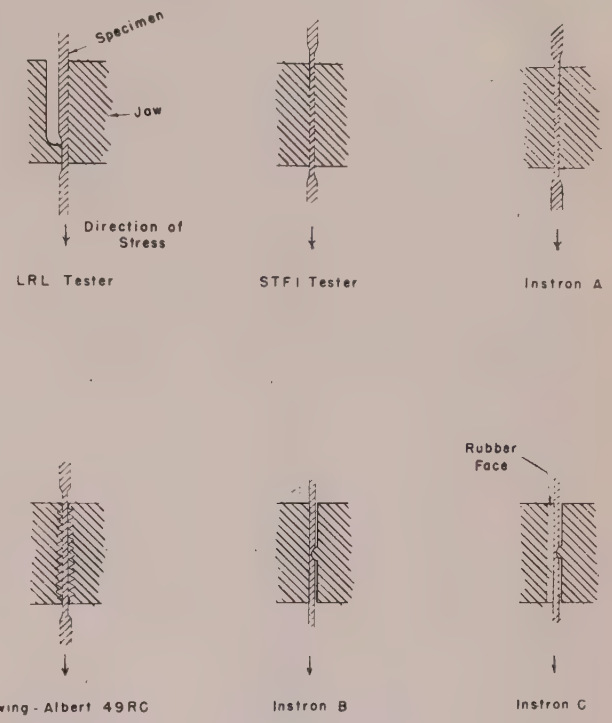

Fig. 15. Jaw designs used in the various testers

Table V. Reproducibility Data

Type of tester	*95% Confidence limits in standard measure for Coating raw-stock, %*	*Charleston kraft MWS, %*	*Clupak MWS, %*	*Creped MWS, %*	*Newsprint, %*
LRL tester	1.90	4.09	4.75	3.74	5.86
STFI tester	2.46	6.74	5.24	3.35	6.89
Instron tester A	2.53	4.16	4.67	2.48	8.09
Thwing-Albert 49RC tester	3.92	2.26	3.83	3.50	7.42
Instron tester B	2.12	4.13	4.61	2.79	7.69
Instron tester C	2.50	3.90	4.20	2.90	4.10
Average	2.57	4.21	4.55	3.13	6.68
	Stretch Data				
LRL tester	3.57	5.72	3.74	5.04	9.85
STFI tester	5.24	6.41	3.15	5.52	9.55
Instron A	7.07	5.49	4.28	5.84	9.73
Thwing-Albert 49RC	6.05	5.93	4.37	5.47	10.57
Instron B	5.15	5.70	4.46	5.12	13.72
Instron C	5.80	7.30	3.80	4.80	10.70
Average	5.48	6.09	3.97	5.30	10.69
	Toughness Data				
LRL tester	5.26	9.96	5.86	6.99	17.97
STFI tester	6.91	12.36	6.46	7.05	14.51
Instron A	8.57	8.67	5.32	4.95	18.90
Twing-Albert 49RC	9.73	7.99	5.17	8.20	18.03
Instron B	7.76	9.65	5.85	7.99	19.04
Instron C	8.60	12.00	5.50	7.00	21.90
Average	7.81	10.11	5.69	7.03	18.39

† The results obtained with Instron tester C should be ignored at this time since their positive bias is caused by a factor other than jaw pull-out.

* The low tensile strength observed when kraft multiwall sack paper was tested on the STFI is due to the use of a 10 mm. wide specimen. This was necessary because of the loading limitations of the tester.

and introduced the greatest positive bias into stretch and toughness determinations.

3. Jaws designed to allow a minimum of contact with the test specimen during testing reduced but did not eliminate jaw pull-out.

4. Papers which had high tensile strengths and low ultimate stretches were most apt to give positively biased toughness results.

Thus, it appears that if accurate stress-strain toughness results are to be obtained, jaws assuring line contact during stress-strain testing must be used.

When the data gathered via Instron C are compared with those gathered with Instron B in Figs. 13 and 14, it can be seen that Instron C gave consistently higher stretch and toughness results than did Instron B despite the fact that little or no jaw pull-out could occur when paper was tested via Instron C because of the type of jaws used. This apparent discrepancy can be explained if it is remembered that Instron C was equipped with an automatic print-out mechanism rather than an autographic recorder. Careful observation of this print-out mechanism during operation showed that its sample break detector was not sensing the exact moment at which specimen fracture occurred. The result was that erroneously high stretch and toughness results were reported. This can, of course, be corrected by equipping the print-out mechanism with a more sensitive break detector.

Because one of the prime objectives of this round-robin study was to determine if the reproducibility limits of the company standard toughness test were appropriate, the 50 specimens of each paper tested on each instrument were divided into five groups of ten specimens each. As a result, five essentially separate tensile strength, stretch, and toughness

Table VI. Data Comparison Tensile Strength Data

Type of paper	*Position with respect to limits*	*LRL tester*	*STFI tester*	*Instron A*	*Thwing-Albert 49RC*	*Instron B*	*Instron C*
			Tensile Strength Data				
Coating rawstock	Above	0	0	0	0	0	0
	Inside	5	5	5	5	5	5
	Below	0	0	0	0	0	0
Kraft MWS	Above	1	0	1	0	0	0
	Inside	4	4	0	1	0	0
	Below	0	4	0	1	0	0
Clupak MWS	Above	0	0	0	0	0	0
	Inside	5	5	5	4	5	5
	Below	0	0	0	1	0	0
Creped MWS	Above	0	0	1	0	0	0
	Inside	2	4	4	5	5	5
	Below	3	1	0	0	0	0
Newsprint	Above	0	3	0	1	1	1
	Inside	3	2	4	4	3	4
	Below	2	0	1	0	1	0
Total	Above	1	3	2	1	1	1
	Inside	19	17	22	22	23	24
	Below	5	5	1	2	1	0
			Stretch Data				
Coating rawstock	Above	1	2	0	0	0	0
	Inside	4	3	4	5	3	4
	Below	0	0	1	0	2	1
Kraft MWS	Above	2	0	0	0	0	4
	Inside	3	2	5	4	1	1
	Below	0	3	0	1	4	0
Clupak MWS	Above	0	1	1	0	0	0
	Inside	5	4	4	5	4	5
	Below	0	0	0	0	1	0
Creped MWS	Above	0	0	0	0	0	1
	Inside	5	5	5	5	3	4
	Below	0	0	0	0	2	0
Newsprint	Above	0	4	0	0	0	3
	Inside	3	1	5	1	2	2
	Below	2	0	0	4	3	0
Total	Above	3	7	1	0	0	8
	Inside	20	15	23	20	13	16
	Below	2	3	1	5	12	1
Coating rawstock	Above	0	1	0	0	0	0
	Inside	5	4	5	5	5	4
	Below	0	0	0	0	0	1
Kraft MWS	Above	0	0	0	0	0	0
	Inside	5	5	5	5	5	5
	Below	0	0	0	0	0	0
Clupak MWS	Above	0	0	0	0	0	0
	Inside	5	5	5	5	5	5
	Below	0	0	0	0	0	0
Creped MWS	Above	0	0	0	0	0	0
	Inside	5	5	5	5	5	5
	Below	0	0	0	0	0	0
Newsprint	Above	0	4	0	0	2	0
	Inside	4	1	4	5	3	5
	Below	1	0	1	0	0	0
Total	Above	0	5	0	0	2	0
	Inside	24	20	24	5	23	24
	Below	1	0	1	0	0	1

determinations were performed on each paper with each instrument. The average 95% confidence limits of each paper for each tester are expressed in standard measure in Table V. From these data it can be seen that, in general, the 95% confidence limits of the tensile strength of all the papers** were slightly lower than those specified by TAPPI Standard T404 m-53 and that those of the stretch of all papers** were slightly higher than specified by TAPPI Standard T457 m-46. On the other hand, the reproducibility of all toughness results** was well below the limits specified by the company standard method. Thus, it would appear that the reproducibility limits of the toughness determination should be reduced from 15 to 10% of the mean toughness for papers of moderate and high toughness. Further proof of this is supplied by the data presented in Table VI which shows that although individual tensile strength and stretch determinations fell outside of the reproducibility limits, 15 and 29% of the time, respectively, toughness results fell outside of the prescribed limits only 6.7% of the time.†

Table VI also shows the importance of errors due to jaw pull-out; for although the frequency with which tensile strength, stretch, and toughness determinations fall above and below the reproducibility limits of the mean values of each test is about equal, it should be

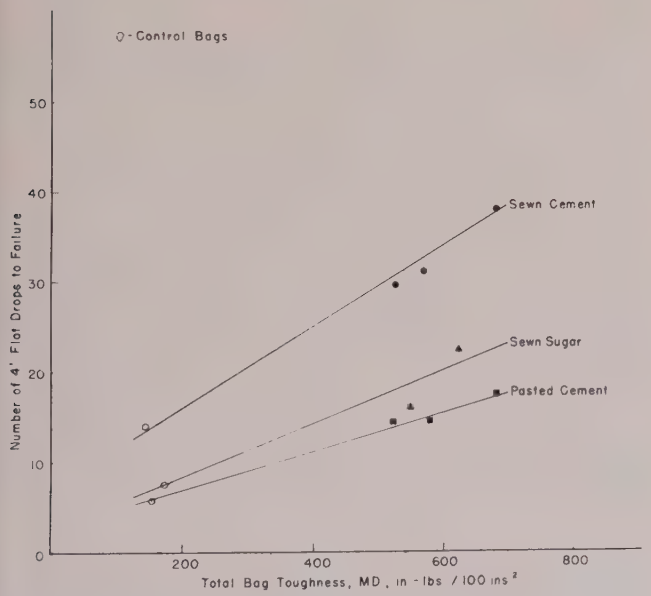

Fig. 16. Total bag toughness versus number of flat drops

noted that the stretch results obtained from Instron B—probably the most accurate data available—fall below the prescribed limits 48% of the time. Thus, if the data presented in Table VI were calculated by reference to the mean stretch and toughness estimated on Instron B, it can be seen that the tendency of all of the testers other than Instron B to give erroneously high stretch and toughness results would be quite evident. This simply re-emphasizes the need for the use of jaws assuring line contact with the test specimen during stress-strain testing.

** The 95% confidence limits of the tensile strength, stretch, and toughness of newsprint were rather high when expressed in standard measure because the absolute values of the mean was so low.

† The fact that the mean of an individual determination falls outside the prescribed reproducibility limits does not necessarily mean that it differs significantly from the mean for its 95% confidence limits may still overlap those of the mean.

The conclusions which can be drawn from this round-robin study are as follows:

1. Although the stress-strain results (tensile strength, stretch, and toughness) obtained on the six testers agreed within the prescribed limits at the 50 replicate level such agreement could not be expected at the 10 replicate level unless scatter due to jaw pull-out, etc., is eliminated.
2. Positive biases introduced into stretch and toughness determinations via jaw pull-out, etc., were statistically significant in certain cases.
3. Jaws assuring line contact with the specimen during testing and sample break detectors of high sensitivity are necessary if accurate stress-strain results are to be obtained rapidly.
4. The limits of variability of the toughness test should be 10 rather than 15% of the sample mean for papers of moderate and high toughness.

THE CORRELATION BETWEEN TOUGHNESS AND BAG DROP TEST RESULTS

Because the stress-strain toughness test was developed as a tool to evaluate Clupak papers, and because multiwall sack paper was the first product

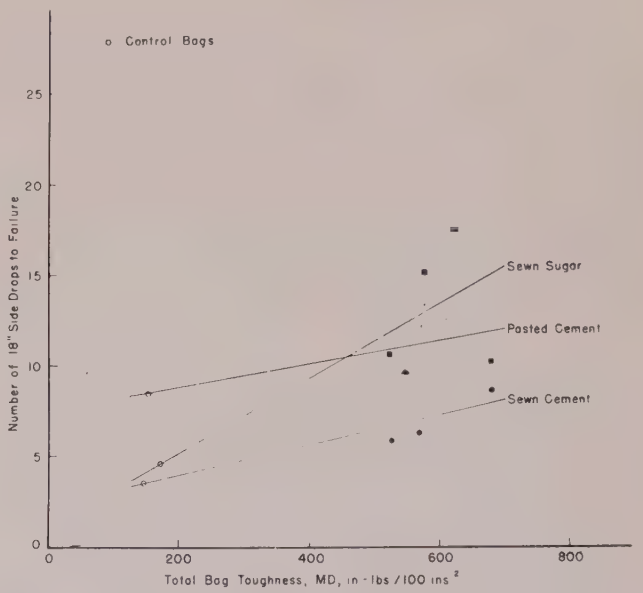

Fig. 17. Total bag toughness versus number of side drops

to be made from Clupak paper, it seems only natural that an attempt has been made to correlate toughness results with the results of the bag drop test—the technique usually considered to be the best test of the quality of bag paper. This was accomplished by forming both sewn and pasted cement sacks as well as pasted sugar sacks from conventional as well as Clupak multiwall sack paper of varying degrees of extensibility. Samples of each type of sack were then selected and the paper of ten were subjected to conventional as well as stress-strain testing before a total of 30 additional sacks were drop tested in each of the three conventional positions. The results of the drop tests were then compared with the various conventional strength tests (tensile, burst, stretch, tear, etc.) and no systematic relation was discovered, but when the drop test data were plotted against total bag toughness* relations such as those shown in Figs. 16, 17, and 18 were obtained.

In Fig. 16 total bag toughness in the machine di-

* The total bag toughness of a multiwall sack is the sum of the individual toughnesses of the plies of the sack.

rection** is plotted against the average number of four foot flat drops required to burst each type of bag. From the plot, two facts are apparent; first, that bags with a high total bag toughness can withstand this particular type of punishment better than bags with a low total bag toughness, and second, that bag design has a definite influence on the efficiency with which a sack uses the inherent strength of a multiwall sack paper.

In Fig. 17 the number of 18-in. side drops required to burst each type of bag is plotted against total bag toughness in the machine direction, and it can be seen that the data are quite scattered. This is caused by the fact that basically the side drop test is a test of the efficiency with which the end closures of a bag are sealed. Thus, although most of the shock energy exerted on a bag during the test is absorbed by the papers of its plies along their machine directions, the bag usually breaks at its end closures. This, of course, causes the scatter but, nevertheless, it is still obvious that total bag toughness correlates fairly well with the results of the side drop test.

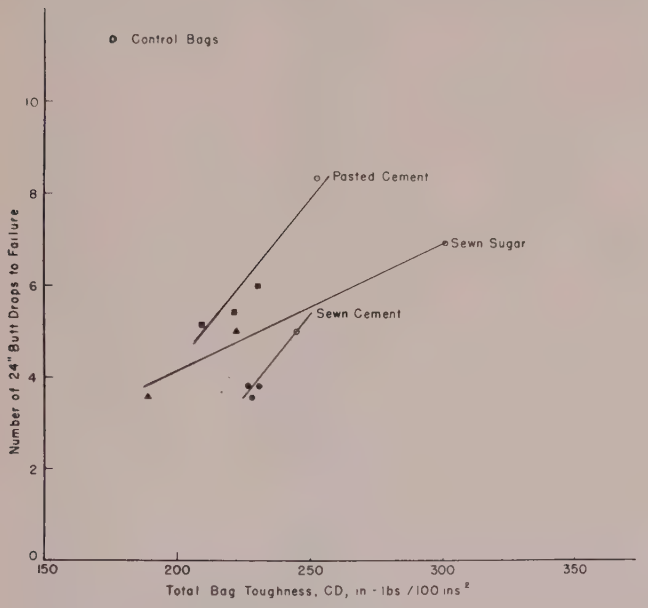

Fig. 18. Total bag toughness versus number of butt drops

Figure 18 contains the plots which were obtained when total bag toughness in the cross-machine direction† was plotted against the average number of butt drops required to burst the various bags. These data again show that total bag toughness correlates well with the results of this drop test.††

It is not the purpose of this paper to prove that total bag toughness correlates well with bag drop test results for it is obvious that there is not sufficient data available to substantiate such a claim. Instead, this information has been presented to show that toughness, which at least theoretically should be closely related to the service strength of paper does show some tendency to correlate well with an accepted use test of at least one type of paper—multiwall sack paper.

** Most of the energy absorbed by a bag during the flat drop test is absorbed by the papers of its plies along their machine direction.

† Most of the work done on a bag during the butt drop test is absorbed by the papers in its plies along their cross-machine direction.

†† The low toughness of Clupak paper in the CD direction has since been remedied.

CONCLUSIONS

The conclusions which can be derived from the work reported are as follows:

1. Although changes in either specimen width or length can introduce biases into stress-strain results (tensile strength, stretch, toughness), the levels used by West Virginia Pulp and Paper Co. in its method of stress-strain testing appear to be reasonable since they lie on relatively flat portions of the various variable versus stress-strain result curves.

2. Changes in the rate of straining at which stress-strain analyses are performed seem to have no significant effect on toughness results (in the range studied) but do introduce a slight bias into tensile strength determinations.

3. Jaws assuring line contact with the test specimen during the stress-strain test must be used if accurate stretch and toughness results are to be obtained via the technique described.

4. There appears to be a systematic relationship between total bag toughness and bag drop test results.

Thus, it appears that the method of stress-strain analysis now being used by West Virginia Pulp and Paper Co. is accurate, reproducible, and essentially unbiased, provided that errors due to jaw pull-out, etc., are avoided and TAPPI has been asked to have an appropriate committee study this method to see if it can be used as a basis for a TAPPI standard method of stress-strain analysis.

ADDENDUM—TOUGHNESS OF PAPER AND PAPERBOARD

The suitability of paper for many end uses depends upon its ability to absorb energy. This energy absorbing capacity of paper can be characterized by the area beneath its stress versus strain curve. This area has the units of force times distance, and when it refers to unit area of paper, is referred to as "toughness." Thus, the "toughness" of paper is equal to its energy absorbing capacity per unit area and is a fundamental sheet property just as are tensile breaking strength and stretch.

Apparatus

The instrument used shall have:

1. Two clamps which make line contact with the test specimen perpendicular to the line of applied stress in the plane of applied stress and hold the specimen in the plane of applied stress throughout the test without twisting, slippage, or jaw pull-out.

2. Means of increasing the strain applied to a specimen at a constant rate until fracture occurs.

3. Means of both measuring and recording the stress and the strain within the test specimen with an accuracy of $\pm 1\%$ during full scale travel.

4. A full scale response of 5 sec. or less.

5. It is recommended that testers on which appreciable numbers of toughness tests are performed be equipped with an automatic integrator and, if so, that it be equipped with a sample break detector of high sensitivity (± 1 lb. at full scale) and an integrator with negligible inertia.

Calibration

Level the instrument accurately in both principal directions, clean all moving parts, and insure the proper operation of all components. Calibrate the stress recording mechanism of the instrument with standard weights via the dead weight loading method. Calibrate the strain recording mechanism with either inside vernier calipers or gage blocks. Calibrate the integrating mechanism via applying a known stress to the stress sensing mechanism over a known distance.

It is recommended that both the stress and the strain recording components of the instrument be calibrated at several points within their range to insure complete accuracy. If either the stress or the strain recording mechanism contains electronic components which are relatively unstable it is recommended that it be recalibrated at least daily.

Test Specimen

The specimens for test shall be strips cut accurately and paralleled to within 0.1 mm. and with clean edges, in each principal direction of the paper, after having been conditioned according to TAPPI Standard T 402 m. When possible, the length shall be 9 in. and the width shall be 1 in. The specimens shall be free from abnormalities, watermarks, creases, and wrinkles.

Procedure

The test shall be made in an atmosphere conditioned according to TAPPI Standard T 402 m. The gage length used during testing will be 5.00 ± 0.02 in. and the rate of straining used shall be 2.00 in. per min. for all papers except those with small amount of stretch (approximately 3.5% or less), whereupon the rate of straining shall be reduced to 1.00 in. per min. or adjusted so that fracture occurs in not less than 5 sec.

Tightly clamp one end of each test strip in the stress sensing jaw after having placed it in the straining jaw to assure good alignment. Next, clamp the other end of the test strip in the straining jaw and begin the loading cycle.

Specimen toughness shall be determined by estimating the area beneath the specimen's stress-strain curve with an automatic integrator, a polar-planimeter, or from the controids of the stress-strain plot. The units of toughness shall be the product of the stress-strain curve coordinates; namely, inches (strain) times pounds (stress).

Reject readings from individual strips if the specimen slips of breaks in or at the edge of the clamps.

The result of each toughness determination shall be recorded to three significant figures when accuracy permits.

At least 10 and preferably 20 specimens cut in each principal direction of the paper shall be tested.

If the mean value of the lowest and the highest reading differs from the average of all the readings by more than 5% for papers having greater than 8 units of toughness and 10% for papers having less than 8 units, more specimens shall be tested until there is agreement within this limit. Thus, in the case of an irregular sheet of paper it may be necessary to conduct a much larger number of tests than 10. An isolated, very high or low result, which is not repeated in duplicate, shall be discarded when a consistent average is obtained without the abnormal reading.

Results

Results obtained on strips cut in the machine direction shall be reported as toughness, machine direction, and the results obtained on strips cut in the cross-machine direction shall be reported as toughness, cross direction.

For each principal direction, the average value of the toughness results shall be reported in inch-pounds per 100 sq. in. The average, maximum, and minimum toughness for each of the principal directions of the paper shall be reported.

A complete report requires, in addition:

1. Maximum and minimum results of individual tests and number of specimens tested.
2. A statement of the length and width of the test specimen as well as the rate of straining used.

Toughness expressed in inch-pounds per 100 sq. in. is calculated from the equation:

$$T = \frac{100A}{LW}$$

where:

T = toughness, in.-lb/100 sq. in.
A = area under stress-strain curve, inches of strain × lb. of stress
L = specimen length, in.
W = specimen width, in.

Reproducibility of Results

Duplicate determinations of toughness from different sets of samples from the same shipment and on different instruments are expected to agree within 10%.

Additional Information

If specimen length is reduced, it is usual to obtain a higher result while decreasing sample width has the opposite effect. Therefore, if sample lengths, widths, or rates of straining other than those specified above are used, special attention should be brought to the fact.

Occasionally, when low stretch papers are strained at high rates the stress developed within the specimen increases at a rate greater than that which can be followed by its autographic recorder. If this is the case, it will be found that the slope of the upcurve during the initial portion of the stress-strain test will be equivalent to that of the downcurve after fracture. If these two slopes are equal or approximately so, there is a good chance that errors due to recorder response have been introduced. Therefore, the test should be repeated using a straining rate (small changes in the straining rate have little effect on toughness, tensile, or stretch results under these particular conditions of testing) which is low enough so that the slope of the upcurve is considerably lower than the slope of the downcurve. (The stretch axis of an autographic recorder may be expanded to aid in detecting difference in the slope of the up and downcurves if necessary.)

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A Simple Laboratory Device for Studying the Trailing Knife Coating Process

G. W. JONES and D. W. QUACKENBUSH

The laboratory instrument described was designed specifically for studying the trailing knife coating process. It can be built at a moderate cost and, being sheet-fed, is economical to operate. Coating is applied by a blade pressing against a raw stock sample, which in turn is affixed to the periphery of a narrow rubber-covered roll. Powered by a spring drive, this roll accelerates to approximately 850 f.p.m. and then decelerates back to zero velocity during slightly less than 1 revolution. Even higher speeds would seem to be readily obtainable. Experimental results obtained with this instrument correlate very well with a commercial operation. It is recommended as a useful tool for coating formulation work as well as for investigating some of the mechanical variables of the trailing knife process.

As an aid to the development of most any coating process it is generally found desirable, and frequently essential, to possess laboratory equipment that simulates the action of the commercial unit being studied. In addition to being invaluable for formulation work, such equipment can also be of assistance in studying certain operating variables of a mechanical nature.

This need was immediately recognized in the present case when the decision was made to investigate the trailing knife coating process. It was felt, however, that the expense of a web-fed pilot unit could hardly be justified, since this equipment might become more or less obsolete once the commercial process had been established. Also, at the other extreme, obsolescence would certainly be the case in the event that this particular coating process should prove incapable of providing the desired results.

The fact that the experimental work was to be carried out on relatively heavy foodboard (up to 26 pt. caliper) also influenced the choice of equipment. It was felt that the problem of supplying base stock to a web-fed unit might prove unduly burdensome to the production department as well as to the laboratory personnel.

In view of these considerations, an inexpensive sheet-fed apparatus was designed and built, its motivation system being patterned after the spring drive mechanism employed by a well-known printability tester. It can readily be said that the utility of the device has even surpassed expectations. It has been indispensable for studying coating formulations and shows good promise of being a useful tool for investigating certain blade variables.

GENERAL DESCRIPTION

Figure 1 is a schematic diagram illustrating the principal components of the unit and their general arrangement. The heart of the system is a roll that is 20 in. diam. and 6 in. across the face. This roll is mounted on a 2-in. shaft which in turn rests in antifriction bearings. The latter are bolted to a sturdy framework fabricated from 8 and 9-in. channel iron (Fig. 2). The roll is rubber covered and also is equipped with suitable clips for attaching the raw stock to its surface.

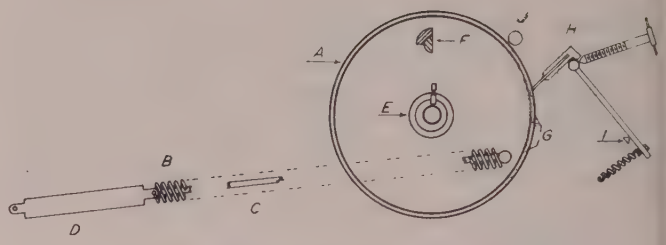

Fig. 1. Schematic diagram of the coater

A. Roll (20-in. diam. by 6-in. face). B. Springs. C. Spring arresters. D. Pneumatic cylinders. E. Cam clutch. F. Release mechanism. G. Raw stock clips. H. Blade holder. I. Lever arm stops. J. Preapplicator.

The roll is caused to rotate by the action of two heavy duty springs, fastened at one end to a shaft near the periphery of the roll and at the other end to pneumatic cylinders. After passing through its cycle of approximately three quarters of a revolution, the roll is prevented from backlashing by a cam clutch, which permits rotation in only one direction. The latter can be disengaged readily in order to return the roll to the cocked position. The final item pertaining to the propulsion mechanism is the release device. Smooth acting and virtually foolproof, this device is operated by twisting a $1^{3}/_{16}$-in. shaft through about one half of a revolution. For reasons of safety it is felt that such an arrangement is far superior to any sort of handle or lever that might be accidentally bumped when the machine is in the cocked position.

The blade arrangement is not particularly complicated, yet it is quite versatile. The holder itself is fashioned from heavy 3-in. angle iron. As shown in Fig.

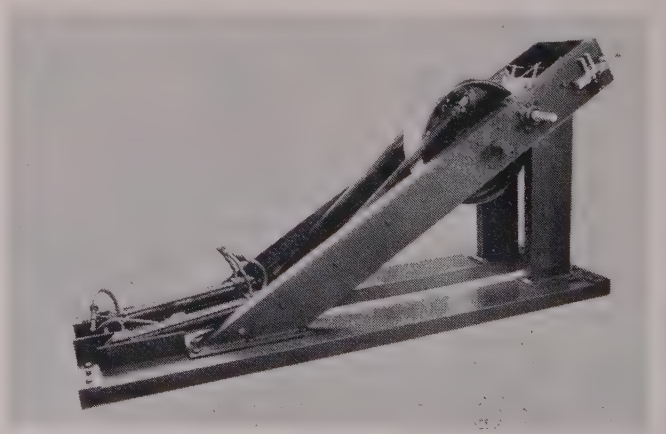

Fig. 2. The sheet-fed laboratory coater

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1, it locks onto a shaft that pivots on bearings. Furthermore, by means of a screw adjustment, the distance between these bearings and the roll surface can be varied. The net effect, then, is that blade angle, pressure and extension can all be varied over wide ranges.

OPERATING PROCEDURE

Raw stock for the laboratory coater is received from the mill in the form of rolls, 36 in. diam. and 6 in. wide. One such roll, cut in the laboratory to 60-in. lengths, will of course provide material for hundreds of experimental applications.

To apply a coating the machine is first pushed into the cocked position by means of the pneumatic cylinders. The release mechanism is then engaged and tension applied to the springs by reversing the travel of the cylinders. Next, the 6 by 60-in. sample is clipped to the surface of the roll, the blade holder locked into place, and a small amount of coating poured into the pond area between the blade and the sheet. Just before the coating begins to flow over the outside edges of the blade (no dikes are used), the machine is tripped through its cycle. The sheet is then released from one of its clips and cut off at the other end immediately below the point where it is still in contact with the blade. Finally, the blade holder is removed and washed, the residual length of sample taken off and thrown away, and the machine returned to the cocked position.

The foregoing procedure, which incidentally is not nearly so messy as might be imagined, requires approximately 3 min. per cycle. Generally, then, coated stock sufficient for a rather extensive testing program can be prepared in less than $^1/_2$ hr.

After the required number of coated strips have been prepared, they are trimmed to dimensions of 4 by 15 in. and finally dried on a Nobel Wood steam drier. Following the customary conditioning treatment they are ready for testing.

Although the foregoing method of operation has proved highly useful for much basic formulation work, it is beset by one notable disadvantage. That is, the depth of the coating pond is far less than that of a commercial unit. Simulation of such a unit is therefore not complete because the contact time between the coating and the base stock is too brief. To remedy this situation a so-called "preapplicator" has been placed approximately 8 in. ahead of the blade. This is simply a 1-in. rod set across the sheet and raised to approximately a 0.015-in. clearance above the surface of the latter. This rod fits into a simple bracket arrangement and is readily removable for cleaning after each coating application. When coating is placed ahead of the rod instead of ahead of the blade, adequate contact time is achieved.

The preapplicator is a relatively recent modification of the machine and has not been studied exhaustively. In view of the considerable data obtained thus far, however, there would seem to be no doubt but what it will prove to be an essential modification in order to achieve good correlation between the laboratory and the mill.

VELOCITY CALCULATIONS AND CALIBRATION

During the process of designing the machine, a method necessarily had to be developed for calculating a spring constant that would produce approximately the desired maximum speed. Furthermore, a theoretical velocity curve was needed in order to determine whether or not the maximum portion of the curve would be flat enough to provide coated samples of sufficient length for testing. The basic equality that was employed for these calculations is as follows:

$$\begin{bmatrix}\text{Rotational energy of the roll at any given instant}\end{bmatrix} = \begin{bmatrix}\text{Initial energy of the fully stretched springs}\end{bmatrix} - \begin{bmatrix}\text{Residual energy of the partially stretched springs at the same instant}\end{bmatrix}$$

This is the same as saying that the rotational energy of the roll is equal to the energy imparted by the spring as it contracts from full extension to some given partial extension. The effects of bearing friction and the friction of the blade against the sheet are thus ignored.

Translated into mathematical terms, the above equation becomes:

$$1/2\ \mathrm{I}\omega^2 = 1/2\ kx_0^2 - 1/2\ kx^2 \qquad (1)$$

where:

I = moment of inertia of the roll (slug sq. ft.)
k = spring constant (lb. per ft.)
x_0 = initial spring extension (ft.)
x = spring extension at any given time (ft.)
ω = angular velocity at spring extension x (radians per sec.)

Equation (1) can readily be transformed into three convenient forms:

$$k = \frac{\mathrm{I}v^2}{R^2(x_0^2 - x^2)} \qquad (2a)$$

$$k \backsimeq \mathrm{I}\left[\frac{v_{max.}}{rx_0}\right]^2 \qquad \text{(Since } x \text{ can be made to approach zero at maximum velocity.)} \qquad (2b)$$

$$v = R\sqrt{\frac{k}{\mathrm{I}}(x_0^2 - x^2)} \qquad (3)$$

where:

r = roll radius (ft.)
v = peripheral velocity (ft. per sec.)
$v_{max.}$ = maximum peripheral velocity (ft. per sec.)

Referring to equation (2b), the maximum spring extension in the cocked position (x_0) and the radius of the roll (r) are both known values, and the moment of inertia of the roll can readily be approximated from direct measurements. Thus a spring constant, k, can be calculated for any preassigned maximum velocity. Then, by means of equation (3), a complete velocity versus extension curve can be calculated. This can easily be translated into a plot of velocity versus position on the periphery of the roll.

The calculations for the original set of springs were made on the basis of 800 f.p.m. maximum velocity. Equation (2b) indicated that a spring constant of 193 lb. per ft. would be required. Since the machine was designed for two springs operating in parallel, two were ordered, each one having a constant of 100 lb. per ft. It was considered desirable to have the constant slightly on the high side in an attempt to overcome possible error due to friction. The theoretical speed curve, calculated by means of equation (3) on the basis of the two 100-lb. per ft. springs, is shown as (a) in Fig. 3. For all practical purposes this curve can be considered identical to curve (b), which was later determined by actual measurement of velocity.

Curve (c) is a plot of velocity versus position for a lighter set of springs. Also, approximately 20 lb. of lead

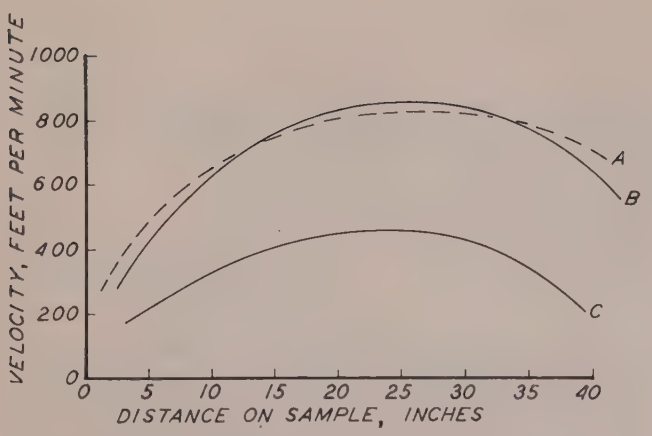

Fig. 3. Velocity versus position curves

A. Calculated on basis of 100 lb./ft. springs. B. Actual for 100 lb./ft. springs. C. Actual for 23 lb./ft. springs.

weights have been bolted beneath the rim of the roll, a technique that is quite useful for making slight changes (either increments or decrements) in the maximum velocity of the roll.

Actual speed curves, such as (b) and (c) in Fig. 3, were derived from photographic measurements that were made as follows: While the room was in total darkness, a strip of 35-mm. photographic film was affixed to the periphery of the roll. Then a fine beam of light from a neon bulb was focused upon the film. When connected to an ordinary 60 cycle a.c. electrical source this bulb emitted a flash twice per cycle, once on the positive side of the voltage surge and again on the negative side. Thus, as the roll was sent through its single revolution, the light beam traced a series of dashes on the photographic film. After measuring the distances between the centers of the dashes, it was then a simple matter to construct a complete velocity versus position curve. It is notable that the arrangement was such that the blade was against the sheet and coating was in the pond when these velocity measurements were made. Hence, the frictional effects present during an actual coating application were faithfully reproduced.

DETAILS OF PARTICULAR INTEREST

The success of this instrument probably cannot be attributed as much to its general design as it can to the ultimate solution of several secondary problems, some of which had not even been anticipated at the start of the project. A number of these problems, as well as other features of interest, are discussed briefly below:

The Cam Clutch. This device would seem to be the only foolproof solution to the problem of arresting the roll at the end of its cycle. It is extremely compact, being approximately the same size and shape as an ordinary roller bearing. The only ingenuity required for this particular application is that of providing a quick disengagement device so that the roll can readily be returned to the cocked position.

The Blade Lifter. Although the cam clutch is very efficient, it still allows a fraction of a degree of backlash, and this has been found sufficient to damage a coater blade quite beyond repair. To remedy this problem an arm has been extended from the blade holder down to a point next to the rim of the roll. A narrow track attached to the rim engages the lever arm toward the end of the coating cycle and acts in such a manner as to push the blade just free of the sheet. It is to allow for this movement that the lever arms of the blade holder are held against stops by springs instead of being rigidly fastened in place (see Fig. 1). Extremely thin coatings tend to run down on the sample as the blade moves away. However, quick action with a cellulose sponge almost always prevents significant damage to the coated sample.

The Spring Arrestors. Originally the two main drive springs were attached at one end to the eccentric shaft on the roll and at the other end to the pistons of the pneumatic cylinders. It was immediately discovered, however, that this simple linkage was not entirely satisfactory. At the termination of a coating application the springs would vibrate violently, and would continue to do so for several minutes thereafter. Despite the fact that the vibratory motion did not appear to contribute adversely to the coating process, the general effect created was not at all that of a smoothly operating scientific instrument.

It was ultimately deduced that the roll, just after passing through maximum velocity, was imparting a whipping motion to the springs, thus setting up the vibrations. The solution seemed to be, then, to form a rigid connection between the eccentric roll shaft and the pneumatic cylinders. When this was done by means of telescopic tubes placed inside the springs, the annoying vibrations were eliminated completely. The operation is now smooth and silent.

Roll Covering. Since the roll does not pass through a complete revolution when a coating is applied, it is permissible to have a seam in the rubber covering. Therefore, a relatively expensive rubber covering job is not necessary. It has been found entirely satisfactory simply to wrap the roll with $^1/_4$ in. thick rubber belting of suitable smoothness and hardness. The ends are secured with stove bolts, and these same bolts also serve to fasten on the clips that hold the sample of raw stock. Damaged coverings are thus easy to replace. Also, this arrangement facilitates an investigation of the effect of roll hardness upon the quality of the coated product.

Blade Honing. Satisfactory coatings could not be applied until a blade was honed in to fit the surface of the raw stock. Since it was not practical to carry out the honing process by making a great many coating applications, a more suitable method had to be developed. The arrangement now used consists simply of a special bracket set next to a steel surfaced roll that is identical to the coater roll. Though the roll is motor driven, experience has since shown that a hand crank would serve admirably. A new blade is inserted in the regular blade holder, which in turn is locked to the bracket of the honer. After the angle and pressure of the blade against the roll have been adjusted, the blade is ground in, using a mixture of coating color and 1000-mesh carborundum grit. Under average pressure conditions a new blade can be honed completely in less than 4 min. Blades that are already ground in but which have received slight damage can be "touched up" in a matter of seconds.

Spring Constants. It is notable that a considerable amount of force may be required to "unlock" the coils of a tightly wound spring. Thus a spring constant measured from the locked position may be quite high. An "initial constant" of this sort does not apply to the

coater since the springs are so installed that they never contract completely.

A true spring constant (and the one that applies to the coater) is determined by first weighting the spring until the coils separate slightly. Then the lineal stretch per unit weight is determined through the further addition of weights. A good deal of difficulty can be avoided if this method of measurement is initially explained to the spring manufacturer.

CONCLUSIONS

The task of designing and constructing this instrument admittedly was undertaken with some misgivings. It was anticipated that there would be considerable difficulty in attaining adequate speed and that the rapid acceleration and deceleration necessarily involved might set up vibrations that would adversely affect the coating application. Fortunately, however, these fears failed to materialize and other minor problems that later arose were solved without a great deal of trouble. The instrument has become so indispensable that it is now difficult to visualize how the laboratory could be of significant service to the mill without it. Its primary attributes might be summarized as follows:

1. It is economical to build and operate.
2. It provides the precision necessary to study some of the more subtle variables of coating formulation.
3. It correlates very well with the mill operation.

An instrument designed generally according to the principles outlined above is highly recommended as a favorable compromise between the one extreme of having nothing but manual drawdown devices, and the other of going to a relatively expensive web-fed pilot unit.

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Gas Chromatographic Analysis of Hydrogen Sulphide, Sulphur Dioxide, Mercaptans, and Alkyl Sulphides and Disulphides

DONALD F. ADAMS and ROBERT K. KOPPE

A gas chromatographic technique is presented for the separation and identification of complex mixtures of volatile sulphur-containing compounds of the type found in kraft pulp digester blow gas and black liquor combustion products. A new series of nine column solvents of the alkyl aryl polyether alcohol type is proposed. A column prepared from Triton X-305, one of the polyether alcohol series having a polyoxyethylene side chain of approximately 30 units, produces the best separation of mixtures containing mercaptans, alkyl sulphides and disulphides, hydrogen sulphide, and sulphur dioxide. Application of this procedure to the analysis of samples containing high ratios of water to sulphur compounds may require a prior water separation before submitting to gas chromatographic analysis.

In the evaluation and control of sulphur losses in kraft pulp production and recovery operations there is need for rapid qualitative and quantitative methods for the determination and identification of sulphur dioxide, hydrogen sulphide, mercaptans, and alkyl sulphides and disulphides in admixture. Available, conventional chemical methods for such mixtures require the use of prior solvent separation, pyrolytic conversion, and/or distillation procedures because of the chemical similarities of many of the components (*4*, *6*, *8*). These results can only be reported by chemical type or total sulphur rather than by individual constituent.

Gas chromatography offers the potential advantages of relatively low cost, simplicity of operation, handling of complex mixtures without prior subgroup separation with suitable solvents, and a more detailed analysis of these mixtures than would be possible by conventional procedures.

Ryce and Bryce (*10*) have reported that the pyrohydrolysis products of dimethyl disulphide (hydrogen sulphide; methyl, ethyl, and propyl mercaptans; dimethyl and diethyl sulphides; dimethyl disulphide and thiophene) can be satisfactorily separated by gas chromatography on a tricresyl phosphate (TCP) column. They found that use of the two-temperature technique of Drew, *et al.* (*7*) greatly reduced the elution time for the higher boiling components of their mixture. Adams and Koppe recently published a preliminary report on the separation of nine malodorous sulphur-containing compounds using Triton X-100 (Rohm and Haas) as a column solvent (*1*). Gas-liquid chromatographic analyses of C_4 and C_5 mercaptans have been reported by Sunner, *et al.* (*13*) and Liberti and Cartoni (*9*). Since this present work was completed, Spencer, *et al.* (*11*) reported the separation of constituents of natural gas odorant mixtures; Coleman, *et al.* (*5*) have identified three alkyl thiols in Agha Jari crude oil on Dow Corning 550 Silicone oil; and Amberg (*2*) has reported the separation of eight thiols, five sulphides and 11 thiophenes on a TCP column.

Since many of the sulphur-containing compounds thus successfully separated are known to be present in the gaseous kraft mill emissions (*8*), gas chromatography should provide a method suitable for the identification of the many compounds present in this type of effluent. In addition to the various sulphides and mercaptans which may be produced during chemical pulping, sulphur dioxide and partially oxidized mercaptans may be present in the recovery furnace gases.

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Table I. Sulphur-Containing Compounds Studied

Compound	*Boiling point, °C.*
Hydrogen sulphide[a]	−82.9
Sulphur dioxide[a,b]	−72.7
Methyl mercaptan[a,b]	5.8
Ethyl mercaptan[a,b]	36–37
Propyl mercaptan	67–68
iso-Propyl mercaptan	58–60
n-Butyl mercaptan[b]	96–98
iso-Butyl mercaptan	88
tert-Butyl mercaptan	62–65
n-Amyl mercaptan	126
iso-Amyl mercaptan	119
n-Hexyl mercaptan	149–50
Dimethyl sulphide[a,b]	38
Diethyl sulphide[a,b]	92–93
Dimethyl disulphide[b]	116–18
Diethyl disulphide[b]	152–53
Water	100

[a] Compounds in test mixture no. 1.
[b] Compounds in test mixture no. 2.

The presence and/or identity of partially oxidized mercaptans have not yet been established.

This paper describes a procedure which permits a separation of 16 sulphur compounds, including sulphur dioxide, hydrogen sulphide, 10 mercaptans, and 4 alkyl sulphides and disulphides (Table I).

EXPERIMENTAL

The hydrogen sulphide was prepared in the laboratory. Sulphur dioxide was obtained from a pressure cylinder supplied by Great Western Division, Dow Chemical Co., San Francisco, Calif. The other sulphur compounds were "Eastman" or "Practical" grade chemicals purchased from Distillation Products Industries, Rochester, N. Y. In addition to preparing chromatograms from the individual compounds, two mixtures were prepared as shown in Table I. This table lists all of the sulphur-containing compounds which were put through the columns producing the best separations of the six-component mixture (mixture no. 1).

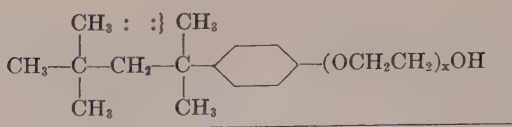
The analyses of the individual compounds and the synthetic mixtures of the sulphur-containing compounds were performed with a Podbielniak Chromacon, Model 9400-3A, three column instrument. Cylinder helium carrier gas, regulated by two diaphragm-type reducing valves in series was used at a flow rate of 50 ml. per min. measured at room temperature. (A higher flow rate was subsequently found to yield less tailing of elution peaks of the disulphides.) The columns, 6 ft. in length and 1/4 in. diam. were prepared from packings consisting of 30 g. of stationary phase solvent to 70 g.

Table II. Gas-Liquid Chromatography Column[a] Solvent Used in Attempted Separation of Sulphur Dioxide, Mercaptans, and Alkyl Sulphides and Disulphides

Cenco Hyvac oil	Polyglycol P-1200
	TCP
Paraffin	Triton X-15 (OPE-1)
DC 550 Silicone oil	Triton X-35 (OPE-3)
DC 550 Silicone oil and 10% stearic acid	Triton X-45
Quadrol	Triton X-114
Pluronic L-61	Triton X-100
Flexol TOF	Triton X-102
Carbitol	Triton X-165 (OPE-16)
Tergitol N-27	Triton X-205 (OPE-20)
Siliclad	Triton X-305 (OPE-30)
Lanolin	Tide[b]
Apiezon M grease	Silica gel[b]
Squalane	Activated alumina[b]

[a] All GLC columns prepared with 30% solvent on 30 to 60-mesh Chromosorb.
[b] 100% 20 to 60-mesh (GSC) columns.

Table III. Octylphenoxyethanol Triton (OPE) Series (Rohm and Haas)



Trade name	*Polyoxyethylene chain length "x"[a]*	*Specific gravity, 25/25°C.*
Triton X-15 (OPE-1)	1	0.985
TCP		
Triton X-35 (OPE-3)	3	1.023
Triton X-45	5	1.040
Triton X-114	7–8	1.054
Triton X-100	9–10	1.065
Triton X-102	12–13	1.071
Triton X-165 (OPE-16)	16	1.080
Triton X-205 (OPE-20)	20	1.088
Triton X-305 (OPE-30)	30	1.095

[a] Products are mixtures with respect to polyoxyethylene chain length, the "*x*" values represent average chain lengths. No information is available on the distribution curves of the polyoxyethylene chain lengths in individual Triton products.

of 30 to 60-mesh Chromosorb (Johns-Manville). The column solvents were dissolved in volatile solvent (usually diethyl ether) and slurried with the Chromosorb. The volatile solvent was then evaporated off at 60°C. under reduced pressure in a Rinco revolving evaporator. Table II lists the various solvents tried. Separation runs were started at an initial column temperature of 30°C. Following elution of diethyl sulphide the temperature was rapidly and reproducibly increased to 100°C. The time at which the temperature was increased was dependent upon emergence of diethyl sulphide and was therefore different for each of the column packings studied.

The separation of hydrogen sulphide, methyl mercaptan, ethyl mercaptan, dimethyl sulphide, diethyl sulphide and dimethyl disulphide on a TCP column as reported by Ryce and Bryce (*10*) was confirmed. It was found, however, that with the TCP column at 30°C., sulphur dioxide and methyl mercaptan could not be sufficiently resolved to permit identification. In addition, incomplete resolution of dimethyl sulphide and ethyl mercaptan was obtained with the TCP column. A wide variety of types of column solvents were then screened in an effort to discover a solvent which would permit a suitable separation of these two compounds and still retain adequate resolution of the other sulphur-containing compounds.

The first satisfactory separation of the constituents of the six-component mixture (Table I) which included sulphur dioxide and methyl mercaptan was obtained using a column of 30% Triton X-100 (Rohm and Haas Triton series of alkyl aryl polyether alcohols) on 30 to 60-mesh Chromosorb. All of the additional solvents examined, with the exception of the Triton series, failed to yield satisfactory results on the six-component test mixture. Resolution of the mixture was markedly improved by use of Triton X-100, the first of the Triton type solvent to be used.

Triton X-100 is one of the nine-membered homologous series of compounds, Table III, and has an average polyether alcohol side chain in the range of 10 to 11 (—OCH_2CH_2—) units in length. Because of the successful separation obtained with this solvent, the Rohm and Haas literature (*14*) was examined to determine the chemical and physical properties of the Triton series.

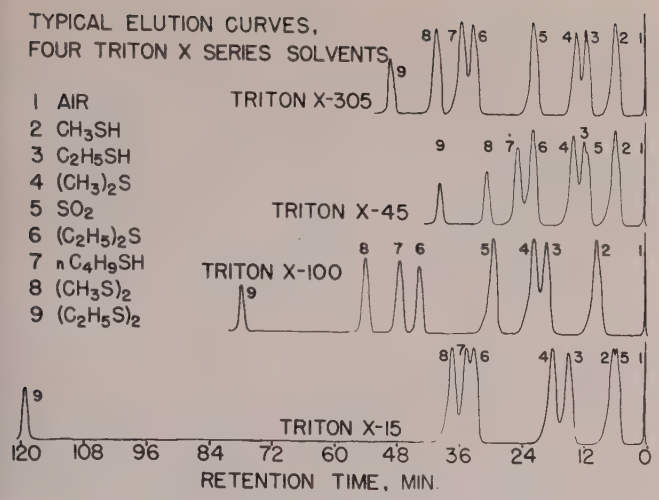


Fig. 1. Typical elution curves, four Triton X series solvent

The water solubility of the nonionic Triton series compounds is dependent upon the hydrophilic character of the ether linkages in the polyoxyethylene chain. These ether linkages are readily hydrated at room temperature and the water solubility of the individuals of the series is related to the number of such linkages present. The compounds listed in Table III, in order of increasing side chain length, are thus also listed in order of increasing water solubility. Hydration forces are reduced by increasing temperature, thus water solubility is greatly reduced at elevated temperatures. Mineral acid solubility of the Triton series is related to its weakly cationic nature, forming oxonium salts with mineral acids. This ability increases with increasing side chain length. Aliphatic hydrocarbon solubility is generally the reverse of water solubility. Triton X-45 demonstrates unusual aliphatic hydrocarbon solubility. This property was later noted experimentally, Fig. 1.

These variations in the properties of the Triton nonionic surface-active agents (*14*) indicated the possibility of controlling the emergence time for sulphur-containing compounds, such as sulphur dioxide without materially affecting others by judicious selection of the side chain length. Similar control of the emergence of water appeared possible through selection of the side chain length and/or the column temperature.

Therefore the relationships between the length of the polyoxyethylene side chain and the emergence time of the sulphur-containing compounds under study (Table I) was examined experimentally. Columns containing 30% by weight on 30 to 60-mesh Chromosorb of each of the additional members of the Triton family were prepared and separations run using the six and eight-component mixtures with and without water.

RESULTS

A series of chromatograms showing the separation of an eight-component mixture (Table I) on four typical Triton columns, using the two-temperature technique, is presented in Fig. 1. Comparison of the elution times for the various components of this mixture indicate that the varying properties of the members of the Triton series have a significant effect upon the rate of elution of (1) sulphur dioxide, (2) mercaptans and alkyl sulphides, and (3) water. From the known properties of the Triton series it had been predicted that the

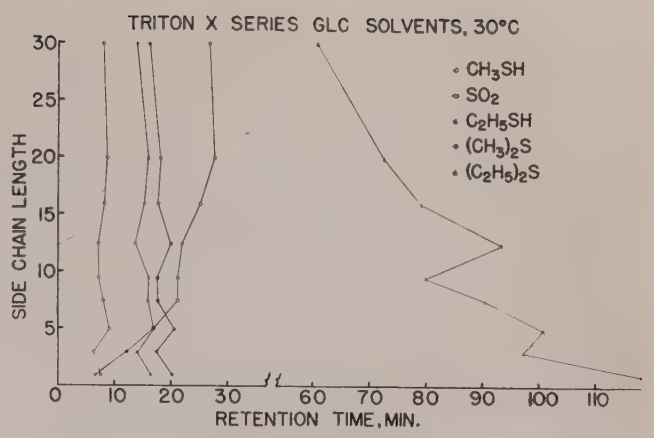


Fig. 2a. Relation of retention time and Triton X side chain length

longer the solvent side chain (1) the longer would be the sulphur dioxide retention time, (2) the shorter the mercaptan and sulphide retention time, and (3) the longer the water retention times. These relations were experimentally substantiated and are shown graphically in Figs. 2a and 2b. The reported anomolous solubility behavior of Triton X-45 with respect to aliphatic hydrocarbons was noted in the two-temperature run. At 30°C., the emergence times for aliphatic sulphur-containing hydrocarbons were in line with the changing properties of the Triton series. With the two-temperature technique it was found, however, that Triton X-45 gave the shortest elution time of the entire series. This solvent is not recommended for use with mixtures containing sulphur dioxide and methyl mercaptan, but can be used to advantage in the absence of sulphur dioxide.

One unexpected relationship was noted, however, as the Triton side chain was increased. Although the retention times for the mercaptans and sulphides tended to decrease as the side chain length was increased, the resolution between ethyl mercaptan and dimethyl sulphide appeared to be somewhat enhanced. This phenomenon permitted us to take advantage of the longer side chain Triton X-305 solvent for decreased over-all retention time for the sulphur-containing alkyl organics, adequate separation of sulphur dioxide and reduced interference from water while obtaining maximum resolution for ethyl mercaptan and dimethyl sulphide.

Following the elution of diethyl sulphide at 30°C. the column temperature was increased to 100°C. to speed

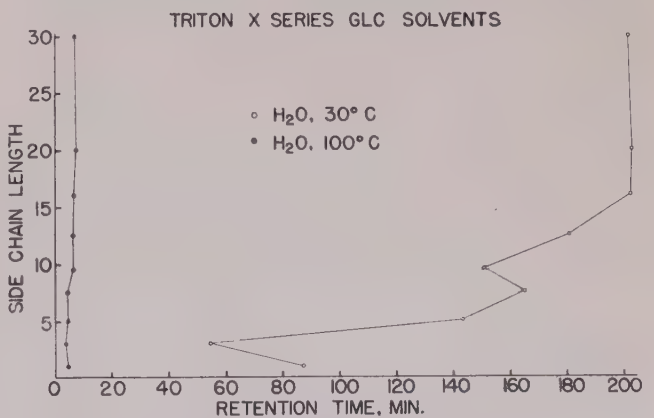


Fig. 2b. Relation of retention time and Triton X side chain length

Table IV. Relative Retention Times for Sulphur Compounds Eluted from Five Column Solvents

Substance	TCP, °C. 30	TCP, °C. 100	Di-n-butyl phthalate, °C. 30	Di-n-butyl phthalate, °C. 60	Di-n-butyl phthalate, °C. 100	Triton X-45, °C. 30	Triton X-45, °C. 100	Triton X-100, °C. 30	Triton X-100, °C. 100	Triton X-305, °C. 30	Triton X-305, °C. 100	Triton X-15, °C. 30	Triton X-15, °C. 100
H_2S	...	0.18	0.07	0.10	0.20	...	...	0.12	...	0.09	0.41	0.06	...
CH_3SH	...	0.54	0.40	0.47	0.52	0.54	...	0.45	...	0.56	0.64	0.46	0.83
C_2H_5SH[a]	...	1.00	1.00	1.00	1.00	1.00	...	1.00	...	1.00	1.00	1.00	1.00
SO_2	...	...	0.43	...	...	1.00	...	1.32	...	1.93	.95	0.40	...
$(CH_3)_2S$	...	1.73	1.15	1.10	0.76	1.21	...	1.09	...	1.16	1.00	1.23	1.42
$(C_2H_5)_2S$	...	4.50	...	5.18	4.04	5.97	...	4.99	...	4.39	2.36	7.24	4.83
n-C_3H_7SH	...	...	...	...	...	...	...	...	...	1.99	1.49	...	1.75
iso-C_3H_7SH	...	...	...	...	...	...	...	...	...	0.96	1.00	...	1.83
n-C_4H_9SH	...	5.18	...	6.12	4.60	...	...	...	...	...	2.76	...	2.67
iso-C_4H_9SH	...	...	...	...	...	...	...	...	...	...	2.17	...	2.83
tert-C_4H_9SH	...	...	...	...	...	...	...	...	...	1.02	1.13	...	2.42
n-$C_5H_{11}SH$	...	...	...	...	...	...	...	...	...	...	5.25	...	7.92
iso-$C_5H_{11}SH$	...	...	...	...	...	...	...	...	...	...	4.07	...	5.83
n-$C_6H_{13}SH$	...	...	...	...	...	...	...	...	...	...	9.51	...	14.33
$(CH_3S)_2$	...	8.14	...	10.90	7.88	...	...	...	...	...	5.71	...	10.83
$(C_2H_5S)_2$	...	25.23	...	44.82	25.64	...	...	...	...	...	13.32	...	27.67
CH_3COCH_3	...	1.32	...	...	...	1.86	...	2.16	...	2.20	1.49	...	2.33
H_2O	...	1.73	...	...	...	8.47	...	9.42	...	14.67	4.78	...	...
K for C_2H_5SH	145.7	13.8	122.4	47.4	12.9	119.3	...	115.6	12.0	102.6	8.9	109.9	6.9

[a] Standard solute.

up the elution of the remaining compounds. Using this technique with the Triton X-305 column required a total elution time of 49 min. This compares with an elapsed time of 73 min. on a column of Triton X-100, 119 min. on Triton X-15, 38 min. on Triton X-45, 130 min. on di-*n*-butyl phthlate, and 47 min. on TCP. Triton X-305 gave much better resolution of the lower boiling components than did the TCP. As previously stated, Triton X-45 could be used for separation of mixtures devoid of sulphur dioxide.

Ambrose, *et al.* (*3*) have recently recommended that gas-liquid chromatographic retention data be given in terms of relative retention times for at least two temperatures together with the partition coefficient of the standard solute. Ethyl mercaptan has been used

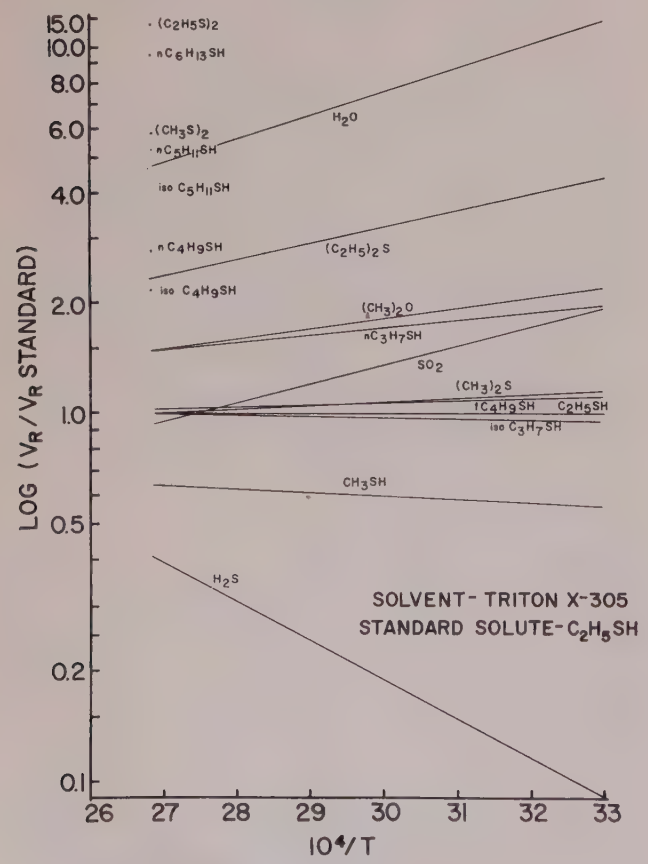


Fig. 3. Effect of temperature on relative retention

throughout our studies as the standard solute in the calculation of relative retention times. Lists of the relative retention times for the various sulphur compounds eluted from (1) TCP, (2) di-*n*-butyl phthlate, (3) Triton X-45, (4) Triton X-100, and (5) Triton X-305 are given in Table IV. In addition, a plot of the logarithm of the relative retention times versus $10^4/T$ for the members of the Triton X series is shown in Fig. 3. Comparison between Fig. 2a-b and 3 shows that there is a greater interaction between the Triton column solvents and sulphur dioxide than is generally shown between the Tritons and the other sulphur-containing compounds studied.

The partition coefficient (3) is expressed as:

$$K = \frac{F_c f \rho_c t}{w}$$

$$K = \frac{\text{(He flow, ml./min.) (press. corr. factor)}}{\text{(density of solvent, g./ml.) (retention time, min.)}} \Big/ \text{(weight of solvent, g.)}$$

The base line curve produced by the Podbielniak Chromacon when operated on an increasing column temperature cycle during the elution of a given sample exhibited some objectionable drift during the temperature rise. This phenomenon was undoubtedly due to changes in the balanced relationship between the thermistor detectors over the range of column temperatures used. This base line drift could be minimized by thermostating the detector cell in a separate constant temperature housing maintained at a constant temperature while the column temperature is raised in its own compartment.

Continuous increase in column temperature throughout the entire course of an elution run has been used by several investigators with success when the sample mixtures contain compounds with widely divergent boiling points (*12*).

Examination of the relationships between the log of the relative retention values and $1000/T$ illustrated in Fig. 3 graphically portrays the difficulties encountered when the continuous temperature rise technique was used on the eight-component mixture. Because of the abnormalities in the relationship between the log $V_R/V_{R\ \text{Standard}}$ and $1000/T$ for hydrogen sulphide, methyl mercaptan, iso-propyl mercaptan, ethyl mer-

captan, and dimethyl sulphide, it could be predicted that an increasing column temperature during the elution of these compounds would result in a reduction in the degree of resolution obtainable. Not until dimethyl sulphide is eluted are there sufficient differences in $V_R/V_{R\ \text{Standard}}$ to permit use of an increasing column temperature. Thus the two-temperature technique which was used throughout these studies provides the best method of obtaining adequate resolution in a minimum of elution time.

CONCLUSIONS

A method has been developed which may be used to separate complex mixtures containing hydrogen sulphide, sulphur dioxide, mercaptans, and alkyl sulphides and disulphides. A new type of column solvent has been used which permits wide selection of the emergence times for water, acidic compounds, and alkyl organic sulphur-containing compounds. Triton X-305 (OPE30), an alkyl aryl polyether alcohol having a polyoxyethylene side chain of approximately 30 units, combined the advantages of resolution of mixture components, with minimum emergence time for the Triton series, and maximum water retention time to prevent masking of the significant areas of the elution curve.

Direct application of this procedure to the analysis of samples containing high ratios of water to sulphur compounds may require a prior water separation procedure in order to obtain a sufficient concentration of the sulphur compounds within the maximum sample volume usable with a given instrument.

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A New Technique for the Evaluation of Paper Mill Toxicants

ARTHUR M. STERN

A laboratory procedure for the realistic screening of paper mill biocides is described. The test involves a serious attempt to simulate, as closely as possible, conditions in the paper machine system while maintaining adequate simplicity in design so as to facilitate the practical running of a large number of tests. The contact environment is mill white water and the inoculum is a mixture of organisms (10^6 to 5×10^7 per flask) in a slime sample taken directly from an area of accretion in the paper machine itself. Measurement of effectiveness is made by weighing the amount of slime adhering to receptor sticks placed in the contact medium for a period of 48 hr.

THE two most dangerous concepts in the lexicon of slime control are "extrapolation" and "tradition." Extrapolation is the nebulous process by which a recommendation for the treatment of one mill or a single machine is applied to many such units. Tradition implies that the approaches applicable in the mid-twenties cannot be fruitfully altered in a modern paper mill. Both attitudes preclude the use of scientific thought and technology which are integral parts of modern industrial growth.

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Many testing methods that are used routinely in evaluating the efficacy of biocides are archaic and inadequate. Some have been adopted from procedures which were originally designed to provide information in fields totally unrelated to the paper industry. The phenol coefficient type of analysis is an example of how a reasonable test for one purpose has been misapplied in another field. Rideal and Walker (*3*) originally designed the phenol coefficient test as a means of relating the strengths of phenoliclike compounds to a standard phenol solution. Several groups are now using modifications of this test routinely for selecting paper mill toxicants. The most obvious defect in this application is the fact that the test involves the use of pure cultures in an artificial nutrient environment. Overlooked entirely are the interactive involvement of many microbial species and the somewhat selective affinities of various chemical moieties for the complex organic constituents of the medium.

Plate counts have also been employed as a means of diagnosing critical slime conditions and evaluating specific biocide efficiencies. It does not necessarily follow that a high bacterial count on an artificial medium is indicative of a dangerous slime condition. As a matter

Fig. 1. Obtaining slime samples

of fact, Shema, Anderson, and Appling (*5*) found that a very poor correlation exists between the total population of a white water system and machine condition. This has been corroborated by our own experience. In many instances, the predominant types of microorganisms that appear on a nutrient agar plate probably play no role in the formation of capsular accretions.

There have been several toxicant evaluation tests that have been designed to overcome the objections raised to the use of the traditional methods described above. King (*2*) and Appling, McCoy, and Shema (*1*) employed white water as the contact environment and the natural flora associated with it in their tests. However, survivor counts on nutrient agar plates were again used as the criterion of efficacy. Shema, Anderson, and Appling (*4*) also incorporated the use of a slime measuring unit in the circulating fluid of a paper machine and measured the amount of material that accumulated on the immersed stick. The principles involved here are good but the test's limitations for use as a routine laboratory screening tool is obvious.

The method to be described in the following sections is actually a combination of many other techniques that have been cited in the literature. We feel that it merely represents the beginning of the development of a sound and practical laboratory method for realistically screening paper mill biocides.

PROCEDURE

Obtaining Samples

In order to adequately screen a number of biocides for their effectiveness against a mixed microbial flora found in a specific paper machine system, it is necessary to obtain a sample of the slime mixture itself and a representative sample of the white water coursing past the area of accretion. These materials are obtained in sterile containers (Fig. 1) so as to minimize the influence of as many external factors as possible during the running of the test.

Preparation of the Inoculum

A portion of the inoculum is weighed into a sterile 125-ml. Erlenmeyer flask containing glass beads and a measured volume of sterile water and fitted with a rubber stopper. A semblance of homogeneity and dispersion of clumps is achieved by shaking the mixture for 1 hr. at 230 r.p.m. It is then passed through a coarse filter to remove the glass beads and resistant clumps.

Although absolute standardization of the inoculum is not necessary since several series of controls provide a suitable means of comparing successive laboratory runs, we had standardized our inocula during the initial stages of our investigations. This was done by means of plate counts and/or turbidometric determinations. Both methods are open to serious criticism but, of the two, the plate count technique is undoubtedly the most accurate.

In preparing the inoculum in the manner described, the population in our contact environment is far in excess of that encountered even in a badly infected machine system. We feel that, by so doing, we are able to subject our biocides to a rigorous screening and are thus provided with a safety margin. In short, if a compound is capable of exerting an inhibitory influence on the large numbers of microorganisms encountered in our tests, there is an excellent probability of success on the machine itself.

Preparation of Media

A. *Contact Medium.* The contact environment, as noted above, consists of both solids and fluids in white water passing through the area from which the slime sample is obtained. If one is merely interested in the slime-building potential of the machine micro-flora, this medium would be distributed to sterile reaction flasks (to be described in a later section) with no additional treatment. However, for routine screening programs, the medium should be sterilized—especially if the white water population is high relative to that of the inoculum. This occurs when an accretion is both chemically and biologically induced as determined by microscopic examinations, plate counts, and chemical tests.

The white water is separated into two fractions either by centrifugation or filtration. The solids are heat sterilized in the autoclave while the fluid fractions are passed through Seitz bacterial filters so as to preserve their chemical integrity. The recombined sterile fractions then constitute the contact medium. In our work we have been using 20 ml. of this medium per 125-ml. Erlenmeyer flask.

B. *Recovery Medium.* A recovery medium of nutrient agar is employed to obtain some information with regard to the number of microbial survivors at the end of the test period. This medium (Difco beef extract,

Fig. 2. Microbial survivors after contact with four levels of a biocide. The concentrations for nos. 35, 36, 61, and 62 are 10, 20, 40, and 60 p.p.m., respectively

0.3%; Difco peptone, 0.5%; and Bacto agar, 2.0%) is prepared in flasks, sterilized in the autoclave, and dispensed into sterile Petri dishes. No attempt is made to obtain absolute counts of survivors but, rather, growth densities of populations (Fig. 2) subjected to different biocides are compared with controls and assigned values ranging from 0 (complete inhibition) to 10 (no inhibition). As will be pointed out later, this provides useful supplementary information with respect to the mechanism of action of the screened toxicants.

Preparation of Reaction Flasks

The reaction flasks in which the test is conducted consists, simply, of a series of 125-ml. Erlenmeyer flasks fitted with rubber stoppers. The stoppers are provided with a slit from one side extending to the center through which is inserted a stick which is held rigidly in place down the center of the flask. The space in the slit not occupied by the receptor stick is filled with cotton which serves as an air vent and thus prevents oxygen from becoming a rate-limiting factor in the test. Initially, untreated tongue depressors were used as slime receptors but we soon found that it was necessary to boil them three successive times and then air dry them prior to use. This served to remove inhibitory substances with which the tongue depressors had previously been treated and also increased their porosity so as to shorten the length of time required to run the test. Later, popsicle sticks were substituted for the tongue depressors but the same pretreatment technique was adhered to. These units—flasks, stoppers, sticks, and cotton vents—are assembled and then sterilized in the autoclave.

Setting Up an Experiment

A series of reaction flasks are set up containing contact medium (20 ml.), inoculum (5 ml.), various concentrations of biocides (usually four per compound), and enough sterile water to bring the final volume of each flask up to 30 ml. Several series of control flasks are also employed to which no biocides are added. One half of these are inoculated and serve as positive controls while the other half are uninoculated and serve as negative controls. All flasks are then placed on a shaking machine (Fig. 3) at a temperature corresponding to the machine temperature at the site from which the samples were obtained. The stroke and speed of the shaking machine are then adjusted so as to provide

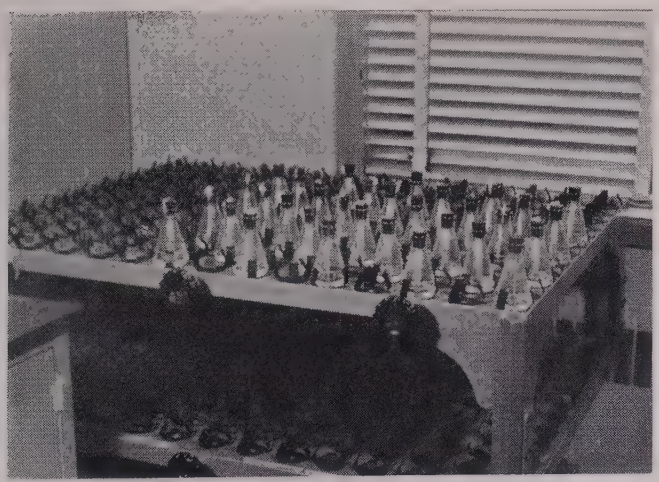

Fig. 3. Flasks on shaker at the end of an experimental run

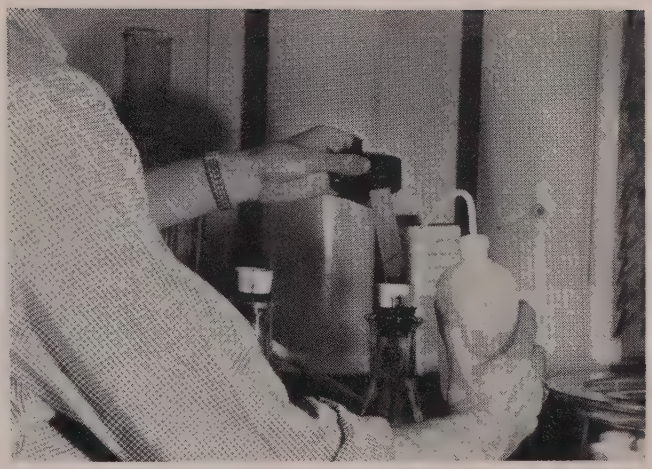

Fig. 4. Elution of slime accretions from receptor sticks

the same degree of aeration (as measured by dissolved oxygen determinations) as that found in the machine area under investigation. We feel that this is quite important in that, in our experience, most of the heavier slime accretions were found at the air-fluid interface. Thus the implication is that almost all of these slime accumulations, at least in their initial stages, are due to the action of aerobes and/or facultative anaerobes. Another reason for maintaining a critical dissolved oxygen level is the fact that many of the toxicants tested are undoubtedly respiratory poisons and, if their mechanism of action involves a competitive displacement of oxygen, they would have to "work hard" to exert their inhibitory influence in machine areas that contain a maximum of dissolved oxygen.

At the end of the incubation period, flasks are removed from the shaker, the accumulated slime on the sticks is eluted with a jet of water into tared Gooch filters (Fig. 4) and oven-dried to constant weight. At the same time, standard loops of the residual fluid in the flasks are streaked on plates of recovery medium. These plates are then incubated for 48 hr. at the same temperature as that employed for the contact environment after which comparisons are drawn between test materials and controls as described above.

Interpreting Results

In the screening procedure described above, we are provided with both qualitative and quantitative information in the form of relative survivor numbers and slime weights, respectively. A careful analysis of this type of data can help produce some of the answers to questions which have consistently plagued those interested in slime control.

The method appears to be applicable to the practical screening of large numbers of biocides against the microorganisms in a particular machine system or, depending upon the position and objectives of the investigator, it can be employed for screening a limited number of toxicants against the microflora of a large number of machine systems. Since the test is conducted in an environment that closely simulates mill conditions, we should be able to arrive at a group of compounds that will prove effective when employed in a mill trial.

We should also be able to reach some decision with respect to use dosages of the compounds of choice. From test results and practical experience we have concluded that the starting point for a mill trial should be

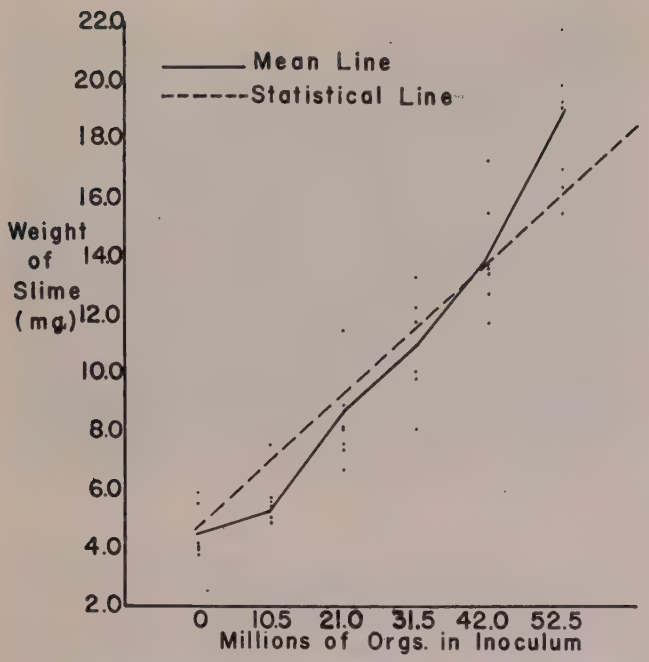

Fig. 5. Relationship of inoculum size and slime accretion

the highest dosage found to be effective in our laboratory which is consistent with economic production. Once control is achieved, treatment levels may be reduced in gradual stages except during those seasons which are traditionally troublesome.

The test should also yield some information pertinent to the mechanisms of action of those biocides employed in the screen. At least four possibilities exist: (1) heavy accretions may be found at all concentrations of a particular compound along with heavy growth on the recovery medium, (2) poor slime accumulation on the sticks as well as low survivor levels on the plates, (3) little slime build-up on the sticks but heavy growth on the nutrient agar, or (4) heavy accretions on the sticks and relatively little growth on the plates. The first case would be indicative of an ineffective compound while the second case would be characteristic of a toxicant that is effective by virtue of its killing or biocidal action. The third case might mean that the compound in question is a good bacteriostat but is no longer capable of exerting an inhibitory influence when diluted out beyond its effective concentration or that the population comprising the inoculum has poor slime-forming properties. A close inspection of the controls should establish the validity of either argument in the latter case. Finally, the fourth case would be indicative of a slime that is chemical and not biological in origin.

DISCUSSION

We recognize certain shortcomings in our screening procedure. Every effort has been made to simulate mill conditions but expediency places a limit on the degree of simulation that it is possible to achieve. The test, as described, is run in the confines of a flask while the mill, with its constant flow of pulp, additives, and fluids is an open system. This merely serves to emphasize the well-known fact that the validity of any test of this sort awaits confirmation by success in the mill itself. Another disadvantage of the method concerns the amount of replication necessary to derive statistically valid results. We have found that eight replicates per biocide level was necessary to provide the degree of reliability desired. However, we also feel that, with time and experience, some refinements can be incorporated into the technique that should reduce this number. Finally, another disadvantage our procedure has in common with other biocide screening methods is the frequency with which tests have to be run. Seasonal fluctuations, furnish changes, and other alterations in the physical-chemical environment of the paper machine lead to a dynamic situation in which the microbial populations are forever changing. These biological shifts are both qualitative and quantitative in nature and make any specific control measure a temporary arrangement.

EXPERIMENTAL

Influence of Inoculum Size and Incubation Time

From Fig. 5 it can be seen that there appears to be a fairly linear relationship between inoculum size and weight of slime collected on the sticks. Deviation from linearity is greatest at the higher inoculum levels. The same situation prevails with respect to the degree of replication—the smallest difference between replicates is found at lower inoculum levels and the spread between replicates becomes greater as the inoculum size increases. Both effects may have similar origins—namely, the greater influence of the logarithmic rate of microbial increase and the repetitive pattern of slime build-up and auto-removal at higher initial population values. In a fixed supportive environment, the increase in the number of microorganisms is logarithmic and the observed effects in the form of slime accretion tend to follow an arithmetic pattern at relatively low bacterial concentrations and then approach a logarithmic rate at higher population levels.

Once accretion begins, it continues until the material on the stick becomes too heavy to be retained in the swirling environment. Strings of material then break

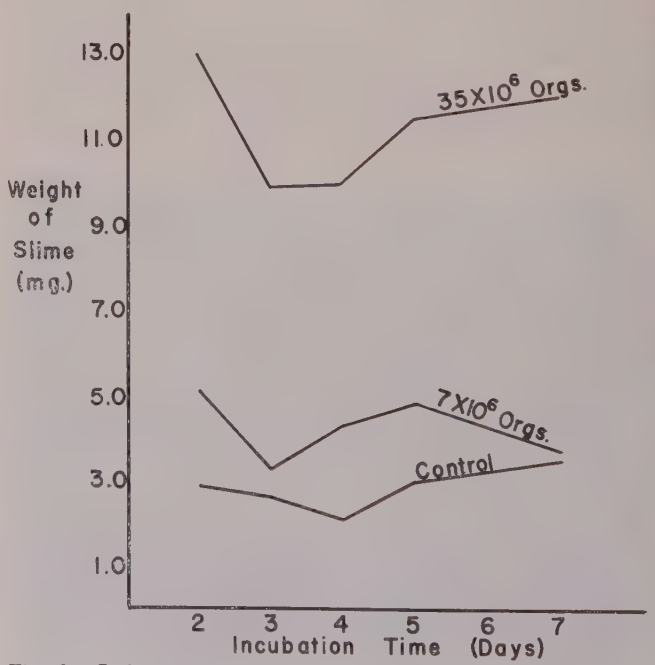

Fig. 6. Influence of incubation time on slime accretion at different inoculum levels

loose which results in the exposure of fresh surfaces on the receptor stick. A secondary slime is then formed until it, too, becomes too heavy and drops off. This sequence can be repeated several times—the frequency of each event during a given incubation period being governed, at least in part, by the size of the starting population. Since the amount of material that drops off the stick appears to be independent of the controlled variables, the degree of replication would be inversely proportional to the frequency of the described events. This is partially illustrated in Fig. 6 wherein the relationship of slime accretion to incubation time is demonstrated at different inoculum levels. In the controls as well as at both inoculum levels employed there is an indication of this cyclic phenomenon. The greatest build-up of slime appears at the end of 48 hr. while the secondary peaks that follow are considerably reduced.

In short, the data indicate that, at higher inoculum levels and shorter incubation times, there is a better development of slime on the sticks. Higher degrees of slime development need not necessarily be realized at the expense of good replication since we have found that reasonably good responses have resulted when the incubation time has been reduced. More specifically, under the experimental conditions, it would appear that the best results have thus far been achieved with an inoculum range from 10,000,000 to 50,000,000 organisms and an incubation period of 48 hr.

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Agricultural Residues as Raw Materials for Viscose-Grade Pulp

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Six agricultural residues were used as raw materials for experimental production of the viscose grade of pulp. All materials were subjected to the same single set of processing conditions in laboratory-scale operations. Yields and chemical characteristics are reported for these experimental pulps, prepared by the prehydrolysis-sulphate pulping process followed by a multistage bleach. Test values for the pulps from wheat straw, corn stover fiber, and bagasse fiber compared favorably with those of six dissolving pulps produced commercially from both hard and softwoods.

Approximately 1.8 billion lb. of cellulosic derivatives, other than paper and board products, were produced in the United States in 1957 (*1*). These products include rayon and acetate fiber, cellophane and other films, cellulose plastics, absorbents, and carboxymethyl cellulose. Rayon and acetate fiber alone accounted for about 1 billion lb. of this total production.

Based on observations reported by the Stanford Research Institute (*2*), it is believed that the annual consumption of rayon and cellulose acetate in the United States will reach 2.8 billion lb. by 1970. Fulfillment of this prediction would mean that consumption since 1950 would have doubled. Manufacturers of high tenacity rayon are actively developing improved products and promoting new markets. For example, special types of strong rayon yarn have been developed that have tenacity and tensile strength comparable to nylon (*3*). Future requirements for raw materials for viscose-grade pulps, therefore, appear to be substantial. Proportionately the price increase for rayon (*4*) over the last 25 years has not been as great as for other commodities. Perhaps this reflects increased efficiency and economy in rayon manufacture.

Softwood dissolving pulps, mainly sulphite, have been satisfactory, but increased use of the softwoods for paper and board has stimulated explorations for other raw materials from which to produce dissolving pulps. Hardwoods were investigated by Richter (*5*) and were found satisfactory. Wood-derived dissolving pulps are prepared by a series of chemical treatments consisting of steam hydrolysis, sulphite or sulphate digestions, and multistage bleaching. Dissolving pulps prepared from wood account for 84% of the rayon and acetate fiber presently produced. The remaining percentage of cellulose used for rayon and acetate fiber is prepared from cotton linters (*6*).

Cotton linter pulp due to its high purity commands a price advantage over regular wood pulp, but the greater cost of linters has also limited their use. The price for cotton linter pulp increased from $172 to $459 a ton during the Korean War while prices for dissolving wood pulps increased from $159 to $185 per ton (*7*).

New raw materials might also have a price advantage if they can be shown to possess characteristics that will make them superior to wood for conversion to dissolving pulps.

Early attempts to use bagasse as a raw material for dissolving pulps failed largely because little or no effort was made to separate the pith fraction from the fiber. In 1928 a pulp mill located at Tuinucu, Cuba, used the De la Roza process for producing dissolving pulp from bagasse. According to at least one source (*8*), this process which did not involve mechanical removal of pith from the bagasse fiber consumed exces-

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sive amounts of chemicals and was otherwise economically unsound. Development of effective methods for pith-fiber separation at the Northern Utilization Research and Development Division (*9*), and by Keller from Louisiana State University (*10*), makes the use of bagasse for dissolving pulps more feasible.

Locus (*11*) produced dissolving pulps from Philippine and Florida bagasse fiber, that had properties comparable to commercial products. He used steam hydrolysis followed by kraft cooking and multistage bleaching. These pulps were tested by a commercial viscose rayon mill and found comparable to wood pulp.

About the time of World War II, the Phrix rayon group in Germany developed a dissolving pulp from straw using a prehydrolysis-sulphate pulping process (*12*). Two sulphate mills (at Kustrin and Hirschberg) were reported to have been ready to produce pulp by this process shortly before the end of the war, but actually did not get into commercial production. A former chemist of a subsidized German mill (*13*) claimed yields of 33% of the cellulose pulp were obtained from wheat straw. Quality of the product, however, was not disclosed.

The objective of the present paper is to report preliminary laboratory studies on the use of six agricultural residues as potential raw materials for the production of viscose-grade pulps. These residues are wheat straw, corn stover, sugarcane bagasse, soybean stalks, seed flax straw, and corncobs (woody fraction). Mechanics of collection have been established for four of these residues, which are already finding some industrial use. Wheat straw is used for corrugating board. Seed flax straw is used for the production of cigarette and other fine papers. Bagasse is used in the sugar mill for fuel and in the manufacture of insulating board and bleached papers. Ground corncobs are used in producing furfural and in surface-cleaning and finishing operations in the metal-stamping industries. The estimated availability of the various residues as well as the production of the principal crops and the acreages involved for the year 1957 are shown in Table I.

Table I. Estimated U. S. Production of Specific Agricultural Residues for 1957

Commodity	*Acreage harvested,[a] 1000 acres*	*Crop production,[a] 1000 bu.*	*Residue*	
			Obtained	*Production, 1000 tons*
Wheat	43,664	947,102	Straw	65,350[b]
Corn	72,656	3,402,832	Stover	114,335[b]
			Cobs	17,014[c]
Sugarcane	289	7,666,000 (tons)	Bagasse	958[d]
Soybeans	20,738	479,841	Stalks	17,274[b]
Seed flax	4864	25,754	Straw	2163[b]

[a] Crop Reporting Board, "Crop Production—1957 Annual Summary," U. S. Dept. Agr., Crop Marketing Service, Cr Pr 2-1(57), 109 pp. (Dec. 17, 1957).
[b] Conversion factors from S. I. Aronovsky, L. E. Schniepp, and Elbert C. Lathrop, "Using Residues to Conserve Resources," Yearbook of Agriculture 1950–51, p. 842.
[c] Ten pounds dry cobs per bushel earcorn, "Corncobs—Their Composition, Availability, Agricultural and Industrial Uses," AIC-bulletin 177, revised, U. S. Dept. Agr. (1953).
[d] Cane production × 0.125. Regional Research Laboratories, U. S. Dept. Agr., 76th Congress, 1st Session, Document No. 65, p. 51.

EXPERIMENTAL

Raw Materials and Methods

All raw materials were of domestic origin. Pith was removed from both corn stover and sugarcane bagasse according to a method developed at the Northern Division (*9*). Only the hard, woody fraction of the corncob was investigated. The term corn stover denotes or indicates the stalk with leaves. Two samples of wood chips—spruce, a softwood, and maple, a hardwood—were obtained from industrial sources for comparative analyses.

The raw materials investigated and the corresponding experimental pulps were analyzed as well as spruce and maple chips and six commercial hard and softwood dissolving pulps.

TAPPI Standard methods (*14*) were used for the ether solubility, alpha-cellulose, and pentosan values. Beta-cellulose was determined gravimetrically and gamma-cellulose by difference. The regular TAPPI cupriethylenediamine (CED) disperse viscosity method at 0.5% concentration of pulp was modified as follows: The pulp was mixed with 12.5 ml. of 0.04% solution of dioctyl ester of sodium sulfosuccinic acid in distilled water and an equal volume of a CED solution (1 *M* copper) to facilitate the dissolving of the pulp (*15*). Lignin was determined by a method previously described by Aronovsky, Nelson, and Lathrop (*16*). MEA cellulose or crude cellulose was determined by a method recently described by Nelson and Leming (*17*).

Analytical results for the raw materials are presented in Table II.

Preparation of Experimental Pulps

Both steam-hydrolysis and sulphate cooking were completed in a 10-gal. jacketed stainless-steel tumbling autoclave. Stainless-steel and stoneware equipment was used in the bleaching of the pulps.

The procedure used to prepare the experimental pulps from each raw material is described briefly:

Nine pounds of air-dry material were put into the digester and the moisture content of the mass increased to 60% (3 parts water: 2 parts dry solids). The damp material was then steamed at 190°C. for 15 min. The hydrolyzed material was washed free from the acids formed during the steaming without removal from the digester. A solution of kraft chemicals (2 parts NaOH: 1 part Na_2S) at the 18% level based on the original dry solids was added to the material in the digester and cooking completed at 170°C. for 2 hr. At the end of the cook after pressure was reduced to 25 p. s. i., the charge was blown from the digester. The resulting pulp was washed free of chemicals, screened, and bleached by the seven-stage treatment described by Locus (*11*).

Yields of bleached pulps were determined from mats formed in a 6 by 9-in. sheet mold fitted with a 150-mesh screen. Yields and chemical characteristics for dissolving pulp from the various crop residues are listed in Table III.

DISCUSSION OF RESULTS

Knowledge of chemical composition of potential and accepted raw materials allows some advance speculation regarding the applicability of new raw materials from which dissolving pulps might be prepared. The data in Table II provide a basis for such preliminary appraisals including six agricultural residues. Pentosan and ash contents, together with substances soluble in

Table II. Proximate Analysis of Various Raw Materials

Material analyzed	Ash, %	Lignin, %	Pentosan, %	Solubility in: Hot water, %	Alcohol-benzene, %	1% NaOH, %	MEA cellulose, %	Alpha-cellulose, pentosan-free, %
Wheat straw	5.2	17.3	29.3	8.3	3.5	38.2	50.9	33.9
Seed flax straw	6.5	16.2	19.9	17.9	6.2	39.1	52.8	37.7
Sugarcane bagasse fiber	2.3	22.3	31.8	2.8	3.0	31.3	60.1	40.5
Corncobs, woody fraction	1.0	14.6	41.8	3.9	2.6	33.5	62.5	31.2
Corn stover fiber	4.8	17.4	23.6	13.5	6.3	13.8	50.0	28.7
Soybean stalks	4.6	17.2	18.6	19.7	5.9	45.0	41.8	27.2
Softwood (spruce)	0.5	27.6	10.6	3.6	2.4	13.9	60.0	39.4
Hardwood (maple)	0.5	23.3	22.6	2.3	2.4	15.7	60.2	39.1

alcohol-benzene indicate that more rigorous treatments would be required for the residues than for the reference woods. The lower lignin contents of the residues, in contrast to those of the woods, should have a compensatory effect, and these should facilitate preparation of dissolving pulps from them. Values for MEA cellulose, alpha-cellulose, and solubility in 1% alkali suggest that yields of viscose-grade pulps from residues would be less than from woods. These opinions are substantiated by data accumulated from pulp yields and by the chemical and physical properties of the residue-dissolving pulps or of commercial-dissolving pulps from wood. It is also evident from the analytical data that these residues require special hydrolysis as well as special cooking and bleaching treatments to remove high ash, pentosans, and the fraction soluble in 1% NaOH solution.

It is not always possible to evaluate completely a dissolving pulp for the particular end use through laboratory tests, but the physical and chemical tests do provide information of value to the viscose producer. Such properties as disperse viscosity; ash; pentosan; alpha, beta, and gamma-cellulose content; and ether solubility determine the types of cellulose derivatives that may be prepared.

The chemical composition of the experimental pulps as presented in Table III shows that several residues have properties comparable to the reference pulps prepared commercially from hardwoods.

Wheat straw pulp has a disperse viscosity value of 10.3 cp. slightly higher than the maximum of the reference pulps. Dissolving pulp prepared from wheat straw was obtained in the greatest yield, 30%, and it had the highest purity and viscosity value among the agricultural residue pulps investigated. In all repects, except ash, the pulp is comparable to the commercial wood dissolving pulps examined. This information on wheat straw, together with the knowledge of its wartime use in Germany, indicates the potential that exists for wheat straw as a raw material from which to prepare commercial-dissolving pulps.

The experimental pulp prepared from corn stover fiber had a disperse viscosity value of 5 cp., slightly below the 6-cp. minimum for the commercial wood pulps analyzed. The stover pulp was essentially comparable to wood pulps in chemical characteristics. The yield of pulp is low, 18% based on separated fiber (depithed material). The yield is even lower, 11%, when based on original stover before pith removal. The recovery value, 51% for alpha-cellulose present, in the original corn stover is also low, but this might be improved through study of conditions of pulp treatment.

Sugarcane bagasse, if considered on the basis of depithed fiber, yields more dissolving pulp, 36%, than does wheat straw; yield based on whole bagasse is only 19%. The recovery of alpha-cellulose from depithed bagasse is 82%, which is comparable to the 84% recovery realized from wheat straw. Chemical characteristics of the bagasse pulp are comparable to those of the wood pulps, but the disperse viscosity value of the bagasse pulp is low.

The recovery value for alpha-cellulose from soybean stalks is the highest, 87%, for the crop residues examined; however, the characteristics of the experimental pulp are slightly inferior to those of the wood pulps.

Table III. Certain Test and Calculated Values for Experimental Pulps from Agricultural Residues and Commercial Viscose Grade-Wood Pulps

Material	Disperse[a] viscosity, cps	Ash, %	Pentosan, %	Ether soluble, %	Celluloses,[b] ash-free: Alpha, %	Beta, %	Gamma, %	Content[c,d] of alpha in original, %	Yield,[d] experimental pulps, %	Actual[d] recovery of alpha-cellulose, %
Wheat straw	10.3	0.55	5.1	0.2	94.7	3.7	1.6	34	30	84
Corn stover fiber	5.0	0.80	2.9	0.1	92.9	6.0	1.1	33 (29)	18 (11)	51 (35)
Sugarcane bagasse fiber	3.5	0.44	3.3	0.3	91.5	7.5	1.0	40 (33)	36 (19)	82 (53)
Soybean stalks	3.6	1.88	3.4	0.6	86.6	11.1	2.3	27	27	87
Seed flax straw	4.0	0.35	3.1	0.7	75.1	22.3	2.6	38	28	55
Corncobs (woody fraction)	3.4	0.41	3.2	0.7	64.0	35.6	0.4	31	14	29
Commercial viscose-grade wood pulps prepared by five manufacturers, 1952 production										
Softwood (1 pulp)	8.4	0.20	2.9	0.3	93.7	3.3	2.8	..	..	..
Hardwood (5 pulps)	6.0 to 9.1	0.09 to 0.37	3.7 to 5.7	0.3 to 0.6	87.8 to 93.6	4.5 to 9.7	1.5 to 2.5	..	..	..

[a] Pulp at 0.5% concentration in cupriethylenediamine.
[b] Corrected for contents of ash and pentosan.
[c] Corrected for content of pentosans.
[d] Figures listed in parentheses represent values based on the original residue including the pith.

Pulps prepared from seed flax straw and the woody fraction of the corncob are inferior to the other crop residue pulps.

Because *only one set of conditions* was used in this exploratory study, it is reasonable to expect that improvement in yields and pulp characteristics could be made with refinement of treatment.

Spectrographic analyses made by the Caterpillar Tractor Co., Peoria, Ill., showed that ash in the final pulp results largely from the quality of water used during formation of the mats. Calcium and magnesium ions could be traced directly to the tapwater used. Ash values therefore can be reduced substantially through use of distilled or demineralized water.

SUMMARY

Experimental pulps were prepared from wheat straw, corn stover fiber, sugarcane bagasse fiber, soybean stalks, seed flax straw, and the woody fraction of the corncob by a single set of hydrolysis-digestion conditions in conjunction with a multistage bleach treatment. Cellulose degradation was more extensive in some cases than in others. These pulps prepared from wheat straw, corn stover fiber, and sugarcane bagasse fiber, appear to have some properties which are comparable to those of the dissolving pulps prepared from hardwoods and should be investigated further. The yield of pulp from corn stover is low. Pulps prepared from soybean stalks, seed flax straw, and corncob material have properties that limit their use. Detailed studies should be made on each material to develop optimal processing conditions and product characteristics. Present technology justifies further consideration of these crop residues as raw materials from which to prepare viscose-grade pulps.

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The authors wish to acknowledge the considerable contributions made by their associates, E. G. Helman, Julia Leming, and O. A. Brandon, in the chemical analyses and by R. L. Brown in the pulping studies. Invaluable assistance was provided by J. A. MacLean and G. Eugene Mortimer, Caterpillar Tractor Co., Peoria, Ill., in conducting spectrographic analyses.

A Study of Some of the Variables in Bleaching Pulp in an Electrolytic Cell

DAVID R. GUSTAFSON

An investigation was made of some of the variables in bleaching pulp in a simple electrolytic cell without the use of a diaphragm to determine how they affected the brightness and strength properties of the bleached pulp. The type of pulp used throughout the tests was unbleached sulphite and the variables studied were temperature, consistency of pulp, concentration of electrolyte, length of bleaching time, current density, and the effect of multistage bleaching. Throughout this study platinum metal was used for the anode of the cell and a stainless steel canister (except for the runs where copper and silver were used as the cathode) served a dual purpose as cell cathode and container. Sodium chloride solution was used as the electrolyte. The results have shown that for maximum brightness and strength, the most important considerations are bleaching temperature and concentration of electrolyte, with the optimum temperature being near 30°C., and a sodium chloride concentration of 5%. From the standpoint of economy it was found that the bleaching should be done in at least two stages, and at a consistency no lower than 3%. The maximum consistency is limited by the ability to agitate the pulp during bleaching. Agitation is very important for uniform bleaching and to prevent degradation of the pulp due to the buildup of chlorine at the anode and sodium hydroxide at the cathode. The use of silver and copper metal for the cathode was found to cause the pulp to become colored due to the action of the chlorine on the metal. The study has shown that with control over temperature, electrolyte concentration and consistency, and by bleaching in two stages, it is quite easy to bleach sulphite pulp (permanganate number 11.9) to a G.E. brightness of 80 points without any noticeable decrease in the strength properties of the bleached pulp.

There would seem to be several advantages in being able to bleach pulp in an electrolytic cell in which the chlorine would be used right after it had been produced. The use of chlorine water systems in commercial bleaching is confronted with a number of problems involving cost. Because of the highly corrosive nature of chlorine, as a liquid or a gas, care must be taken in transportation and storage to see that only noncorrosive containers and equipment are used. For mills that must buy their chlorine from outside sources, transportation can be an appreciable part of the cost.

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Also, since degradation of the pulp is appreciable in high concentrations of bleaching agent, a method of producing chlorine in contact with the pulp would be desirable since it would avoid a high concentration at any one time. Even though chlorine water systems

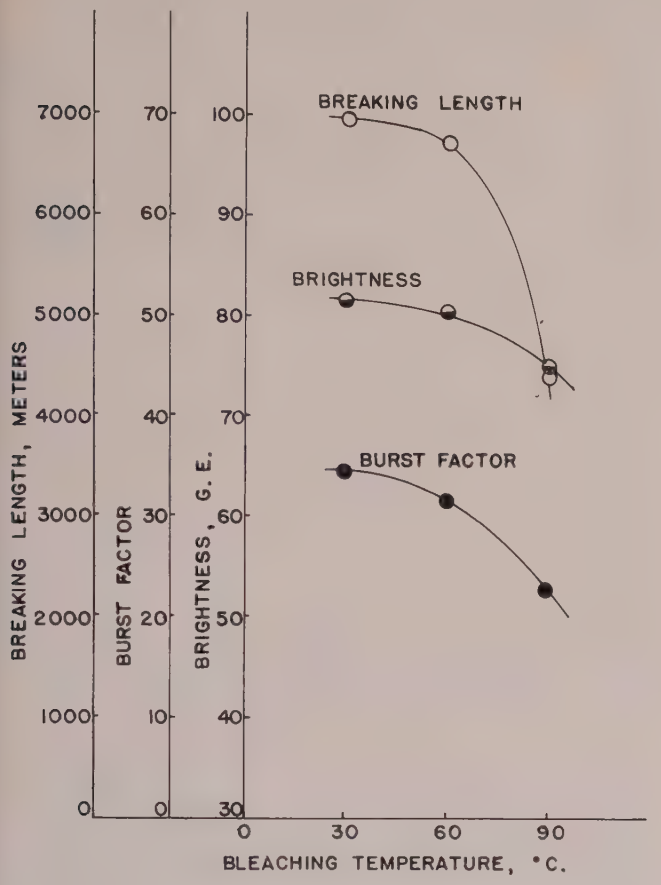

Fig. 1. Bleaching temperature versus physical properties of bleached pulp

have been adapted to continuous processes there are some drawbacks due to limited control.

In consideration of these problems, an investigation was made to study the variables in electrolytic bleaching. Electrolytic bleaching would overcome the problems of transportation and storage associated with corrosive chlorine. It is also possible that electrolytic bleaching might be very adaptable to continuous processes. If so, it would be in line with current trends in the paper industry. One factor that is perhaps most important in any bleaching system is the brightness obtainable for a given cost. This study was made in the hope that it might cast more light on the general nature of electrolytic bleaching.

Previous work by Haines and Kraske (*1*) has shown that bleaching was possible using an electrolytic cell with a diaphragm, a sodium chloride electrolyte, a platinum anode, and a stainless steel cathode. Because of the high cost of platinum, it was hoped that some other material could be found that could replace platinum as the anode.

Preliminary work in this investigation showed that the use of a diaphragm was not necessary, and further, that all metals above platinum on the electromotive series are unsuited for use as the anode. Carbon or graphite could be used for the anode if a permeable wrapping could be found that would prevent the carbon particles from diffusing into the pulp.

The procedure set up for this investigation was to isolate the effect of each of the variables studied by altering the one variable under study while the others were held constant. Following each bleach run tests were made to determine the brightness and strength of the bleached pulp, and on the basis of these tests a final bleach run was made with each of the variables studied held at their optimum value.

DISCUSSION OF RESULTS

The results of this study show that one of the most important factors studied was the bleaching temperature of the stock. The curves in Fig. 1 show that as the temperature of the pulp is increased there is a decrease in fiber strength and brightness. The optimum temperature for both brightness and strength is near 30°C., and as the temperature increases the brightness and strength properties decrease at an increasing rate. In electrolytic bleaching temperature is a factor that

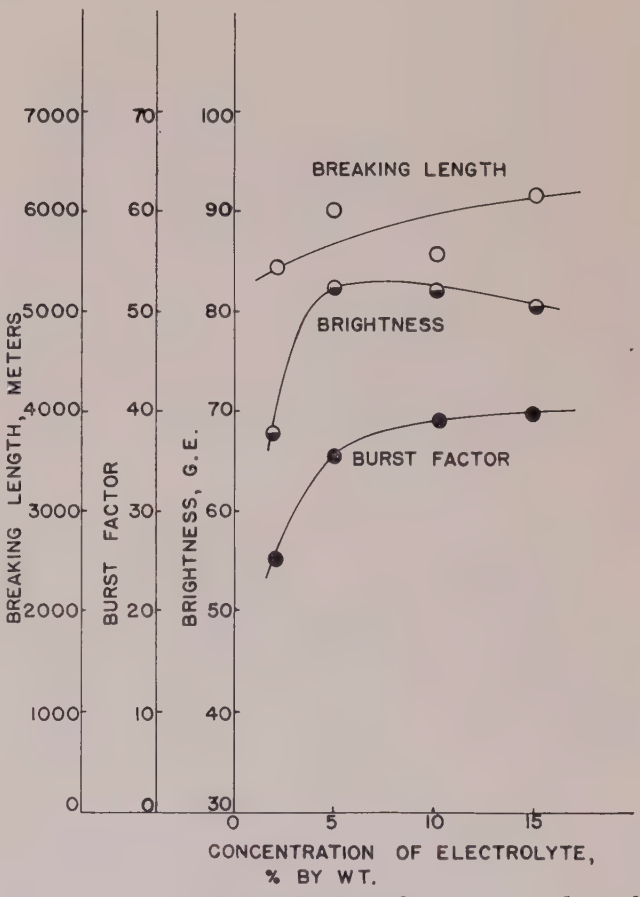

Fig. 2. Concentration of electrolyte versus physical properties of bleached pulp

will have to be taken into account in the design of the equipment, since there is a considerable amount of heat generated in liberating chlorine from the sodium chloride molecule. In this experiment the temperature was controlled by circulating water within the platinum electrode, and by having the stainless steel container in a water bath. In a commerical installation the method

used for controlling the temperature could also be used as a means of heating water for the mill.

The effect of varying the concentration of the electrolyte solution is partially related to the problem of temperature. At low concentrations (2% NaCl) the power requirements of the process are high, and they decrease with an increase in the concentration of sodium chloride. It is desirable to keep power requirements at a minimum, first from the standpoint of cost, and second from the standpoint of the heat generated. Especially with the equipment used in this study in which agitation and temperature control of the pulp was a problem, the higher power requirements were reflected in the low strength and brightness of the pulp bleached in the solution containing 2% sodium chloride. Figure 2 shows the optimum concentration, as far as brightness is concerned, to be near 5%. At a concentration of 10% there was a slight decrease in brightness and the decrease became even more apparent at a concentration of 15% NaCl. However, as the concentration of sodium chloride was increased above 5% the strength properties remained the same or increased slightly. Visual observation of the bleaching runs at concentrations of 10 and 15% would lead to the belief that oxidation of the stainless steel container was a factor contributing to the lower brightness in those runs.

As consistency of the pulp during bleaching was increased from 1 to 3%, the brightness obtainable with a given amount of power increased from 71.2 to 76.7. No apparent difference in fiber strength was noted in the runs made at different consistencies. The factor contributing to the lower brightness at low consistencies is probably due to the lower concentration of reactants in the runs of low consistency. In evaluating consistency as a variable, the factor of keeping costs low would favor as high consistencies as possible. The highest consistency possible with this kind of a process would probably be about 4%, since agitation of the pulp is such an important factor.

Because of the apparent coloring of the pulp by the

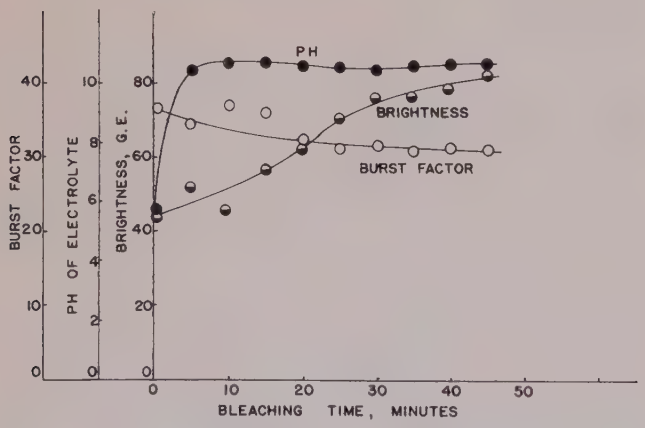

Fig. 3. Bleaching time versus physical properties of bleached pulp

stainless steel container under the conditions discussed, a study of copper and silver for use as the cathode was made. These metals were chosen to be tested, first, because they were available and, second, because they were below hydrogen in the electromotive series. Neither silver nor copper proved at all satisfactory, as the silver gave the pulp a gray color and the copper gave it a blue color. This coloring was probably caused by the reaction of chlorine with the metal.

The results illustrated in Fig. 3 show how length of bleaching time affects the brightness and strength of the pulp, and also the pH of the bleaching solution. The curve of brightness versus bleaching time, though beginning to level off, was still rising at the end of 45

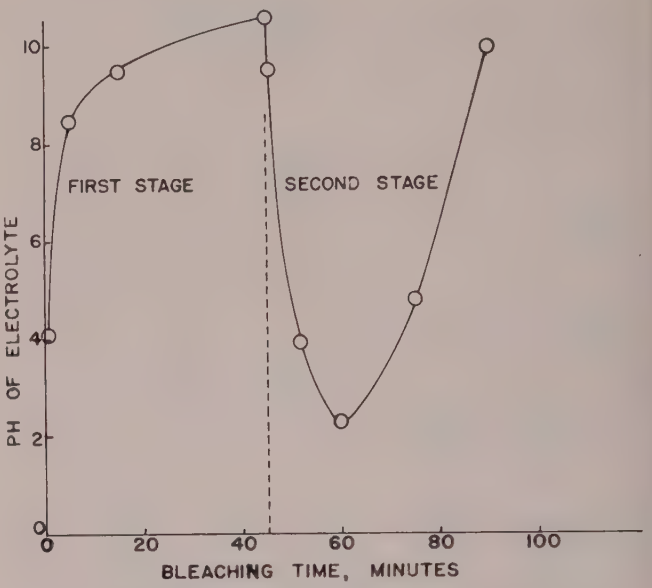

Fig. 4. Bleaching time versus pH of electrolyte for two-stage bleaching

min. so it is quite likely that a higher brightness is obtainable. A sample of pulp taken from the 45-min. bleach run was acidified with a 5% solution of hydrochloric acid. This increased the brightness to 86.3, an increase of 4 points over the brightness obtained during the straight bleaching run of 45 min. This would seem to indicate that the use of a weak acid such as sulphur dioxide solution would be advantageous as an antichlor in raising the brightness of the pulp. The curve of pH versus bleaching time shows that the pH rises very rapidly from 5.8 at the beginning of the bleach run to 10.43 at the end of 5 min. and leveling off near 10.00 to 10.67 shortly after that. The reason for the rapid rise in the pH is thought due to the formation of sodium hydroxide at the cathode, whereas the chlorine liberated at the anode is used in removing the lignin from the pulp. Further studies have shown that it is much easier to bleach in two stages, and the bleached pulp is also much stronger. In this latter procedure the pH takes on quite different values.

There were not any definite differences or trends in the brightness and strength properties of the pulp bleached at various current densities. The pulp used for this run had a permanganate number of 17.0 which was much higher than the permanganate number of 12.2 of the pulp used in the previous runs. The pulp used in this run required about five times as much power in order to bleach to a comparable brightness in one stage. As a result, agitation was much more important, and it is quite possible that inconsistencies in the data are due to inadequate circulation of pulp. Taking these factors into consideration it was thought

that over the range of current densities studied (1.1 to 4.0 amp. per sq. m.)there was no appreciable differences in the bleached pulp resulting from varying the current density.

Two-stage bleaching proved to be a much more efficient method of bleaching than single-stage bleaching

Table I. Electrolytic Bleach Run Sulphite Pulp

Permanganate no. of brown stock	11.9
Consistency, %	3.7
Concentration of electrolyte	5.0
Current density, amp./sq. in.	4.14
Total current × time, amp.-hr.	43.5
Power consumed, watt-hr.	578.5
Weight of pulp bleached, g.	480
Yield, %	86.7
Final brightness	80.25
Brightness after 15 days	78.92

from the standpoint of power cost, brightness obtainable, length of bleaching time, and fiber strength. The results of these tests gave a brightness of 80.2 with 375 amp.-min. for two-stage bleaching while 800 amp-min. were required for a brightness of 74.0 in single-stage bleaching.

Figures 4 through 7 and Table I illustrate the results obtained in the final bleach run. The yield of 86.7% is quite low when compared to yields of about 94% for hypochlorite bleaching in industry (*2*). The low yield may be due to mechanical losses during washing, or inaccuracy in measurement of pulp. Only one run of this type was made so there was no way to check the yield to see if it was accurate. The brightness of 80.2 for the 480 g. of pulp was obtained at a chemical cost of 21¢ ($3.96 per ton) and a power cost of 46¢ ($8.72 per ton) for a total of 67¢ ($12.68 per ton)(*4*). The cost of chemicals for the conventional two-stage chlorine-hypochlorite process would be about $5.00 to $6.00 per ton. There is considerable discrepancy between the theoretical chlorine consumption determined from the permanganate number and the actual chlorine produced as determined by the quantity of electricity passed through the cell. The theoretical bleach consumption as chlorine was 27.7 g. whereas the theoretical chlorine liberated was 57.6 g. Some of the chlorine that was produced may have escaped from the cell as chlorine gas, but it is doubtful that this loss was appreciable. Another reason accounting for the large amount of electricity used may be the formation of sodium hypochlorite and the subsequent liberation of chlorine from the hypochlorite. This involves a valence change of 2, and therefore would require twice as much electricity to liberate a given amount of chlorine. Also there is a possibility that chlorates were formed. The formation of chlorates and the production of chlorine from sodium hypochlorite as just discussed are only possibilities and neither are supported by theory. If the efficiency of the chlorine produced could be increased, both the power and chemical costs could be lowered, or cut in half if the efficiency could be made to approach 100%. The efficiency of chlorine production could also be increased in improved design of the electrolytic cell. Because of the small area of the anode used in this study the chlorine overvoltage was very high. The required voltage, instead of being 13 to 14 v. as it was in this study should be decreased to a value of 4 or 5 v., which is the voltage used in electrolytic cells for the commercial production of chlorine (*3*). This would reduce the power requirements by over one half.

Figure 4 shows how the pH of the bleaching solution changed during the two-stage bleach run. The pH starts at 4.05 and rises rapidly to 8.5 at the end of 5 min. and then continues to rise to 10.65 at the end of 45 min. The reaction taking place is:

$$2NaCl + 2H_2O \rightleftarrows Cl_2 + H_2 + 2NaOH$$

This would seem to indicate that initially the removal of lignin is easy and the chlorine is used up quite rapidly. The excess of sodium hydroxide in solution gives the electrolyte a high pH. Following the first-stage wash the pH was high because of incomplete removal of the bleach solution. As the reaction progressed the pH dropped very fast to a value of 2.31 at the end of 15 min., and then began to rise to a value of 9.83 at the end of 45 min. The initial pH drop after the wash was due to the action of chlorine and hypochlorite bleaching both acting to lower the pH. The combined action of the two types of bleaching outweighs the production of sodium hydroxide. When the pH reaches the minimum

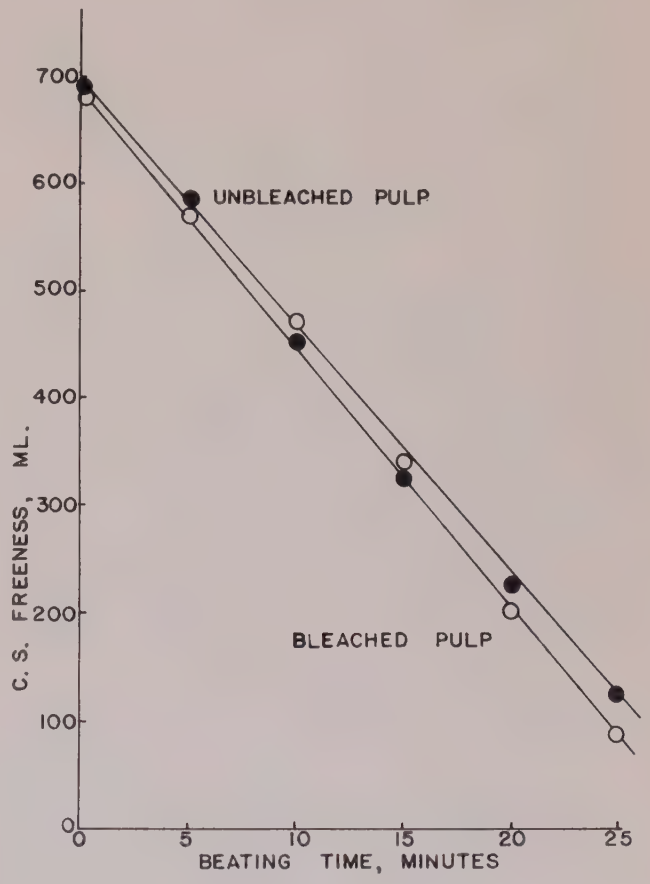

Fig. 5. Freeness versus length of beating time

point the rate of bleaching slows down and the production of hypochlorite increases, which tends to increase the pH. The reaction believed to be taking place in the formation of hypochlorites is:

$$2NaOH + Cl_2 \rightleftarrows NaOCl + NaCl + H_2O$$

There still remains the problem of removing the

soluble lignin from the bleach liquor so that it can be reused. The disposal of this solution would make the cost of this process prohibitive. Two possible methods of removing this lignin from the electrolyte may be (1) chlorination of the lignin by electrolysis in a holding tank after the pulp has been separated from the electrolyte, or (2) using some type of precipitating agent to precipitate the lignins so that they may be removed by filtration.

The curves of Figs. 6 and 7 comparing the physical strength properties of the bleached and unbleached pulp show that there is very little change in the strength of pulp due to bleaching. For a given freeness, the density, breaking length, and burst factor of the bleached pulp was usually higher than for the unbleached pulp. The tear factor of both pulps were about equal. These results indicate that there was very little degradation of the fibers, however there was an increase in fiber bonding due to the removal of lignin. The lack of change in tear factor indicates there was no change in inherent fiber strength, or shear strength, due to bleaching. These results also tend to cast doubt on the accuracy of the yield of pulp for this bleaching run which was quite low, and which would indicate that degradation was extensive. For both

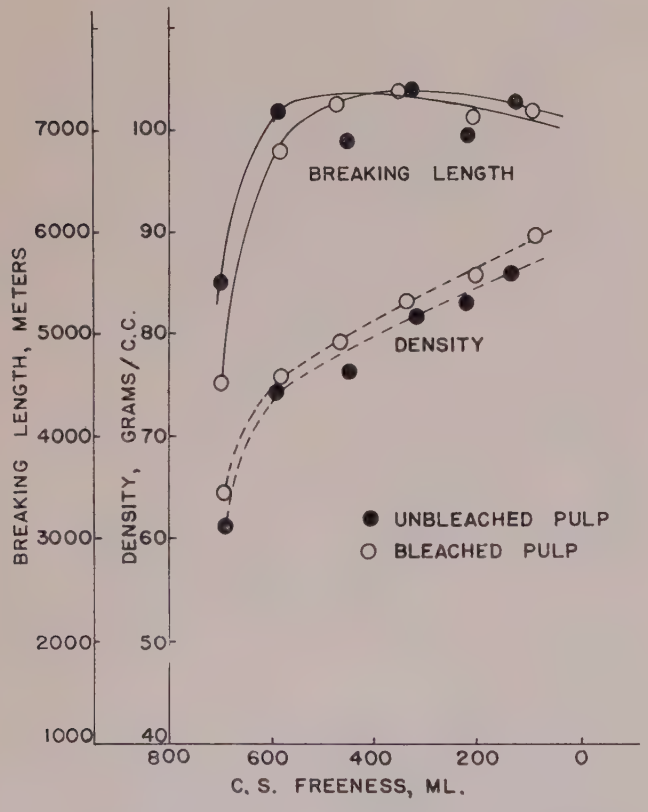

Fig. 6. Freeness versus density and breaking length

pulps the required amount of beating for a given change in freeness was about the same. The color reversion of the pulp after 15 days from the date of bleaching was 1.3 points, from 80.2 to 78.9.

CONCLUSIONS

The following conclusions concerning bleaching pulp in an electrolytic cell may be made from the results of this investigation.

1. Sulphite pulp can be bleached to a brightness of 80 without any appreciable change in the physical strength of the pulp.

2. For maximum strength and brightness the bleaching temperature should be maintained below 60°C.

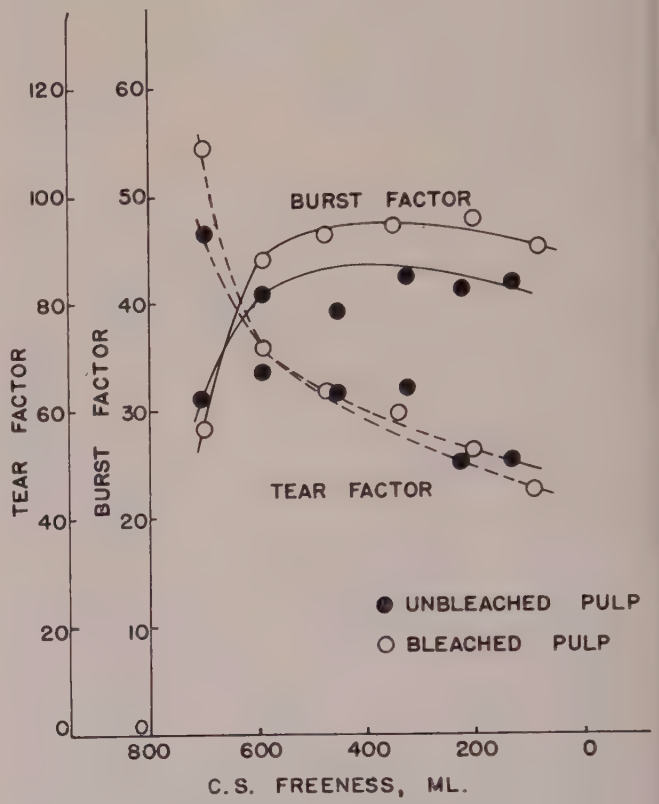

Fig. 7. Freeness versus burst factor and tear factor

3. Low concentrations of sodium chloride (2%) in the electrolyte require an excessive amount of power. This power generates heat within the cell which has a degrading effect on the pulp.

4. Bleaching consistencies should be as high as possible without interfering with the agitation of the pulp.

5. Of the metals tested, stainless steel is suitable for use as the cathode; copper and silver are not. Of the materials tested for the anode, platinum is suitable; all other metals above it in the electromotive series are not. Carbon could be used if it could be wrapped with a permeable material that would contain the carbon particles that flake off during electrolysis.

6. For maximum bleaching efficiency, electrolytic bleaching should be performed in at least two stages.

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A Worthen Braun Award paper presented at the meeting of the Maine-New Hampshire Section of TAPPI held in Orono, Me., Oct. 24–25, 1958.

ASSOCIATION NEWS AND EVENTS

National Conferences

Tenth Testing Conference, Multnomah Hotel, Portland, Ore., Aug. 17–21, 1959.

Fiber Attraction and Bonding Symposium, The Institute of Paper Chemistry, Appleton, Wis., Sept. 9–11, 1959.

Ninth Corrugated Containers Conference, Jung Hotel, New Orleans, La., Sept. 15–17, 1959.

Fourteenth Paper-Plastics Conference, Edgewater Beach Hotel, Chicago, Ill., Sept. 21–23, 1959.

Fourth Deinking Conference, Roger Smith Hotel, Holyoke, Mass., Oct. 8–9, 1959.

Fourteenth Engineering Conference, Penn-Sheraton Hotel, Pittsburgh, Pa., Oct. 12–15, 1959.

Thirteenth Alkaline Pulping Conference, Robert Meyer Hotel, Jacksonville, Fla., Nov. 4–6, 1959.

Other 1959 Conferences

Sept. 13–18	Am. Chem. Society, Atlantic City, N. J.
Oct. 13–15	American Society for Testing Materials, Third Pacific Area National Meeting, Joint TAPPI-ASTM Committee on Petroleum Wax, Sheraton-Palace Hotel, San Francisco, Calif.
Oct. 26–28	Packaging Institute, Statler Hotel, New York, N. Y.
Nov. 9–10	National Paperboard Assoc., New York, N. Y.

Balloting on the Amendment to the Association Constitution

A proposed amendment to simplify the procedure for making future amendments has failed to receive the approval required for adoption. In a mail ballot submitted to the active members in May 481 voted in favor of the proposal, and 571 voted against it. Consequently the amending procedure remains as stated in Article XIII of the Association Constitution.

The Association officers wish to express their gratitude for the interest demonstrated by those active members who participated in the balloting.

Tenth Testing Conference

The Tenth TAPPI Testing Conference will be held at the Hotel Multnomah in Portland, Ore., a large and beautiful city in the center of the fabled Pacific Northwest.

The conference will include outstanding mill visits, many thought-generating papers and a fabulous trip through one of the large tree farms in the virgin forests of Oregon. The conference will include many highlights for your present enjoyment and your future use. The complete program of the conference is presented below for your consideration.

MONDAY, AUG. 17, 1959

Morning and Afternoon

Mill trip with a choice of: Crown Zellerbach Corp., Camas, Wash., or Weyerhaeuser Timber Co., Longview, Wash.

Evening

Social hour followed by Western-style buffet supper (Dutch)

TUESDAY, AUG. 18, 1959

9:00 a.m. Opening Session

Welcome by Mayor Schrunk of Portland
Technical Papers:

1. "Determination of Fiber Length Distribution in Connection with Beating Research," by L. S. Nordman, Oy Keskus-laboratorio-Central-laboratorium Ab, Helsingfors, Finland.
2. "Testing Papers Containing Fluorescent Brightness," by J. M. Patek, Eastman Kodak Co., Rochester, N. Y.
3. "The Tristimulus Method of Fluorescent Brightener Evaluation," by R. S. Hunter, Hunter Associates Laboratory, Falls Church, Va.
4. "Methods for Sampling and Flow Measurement of Pulp and Paper Mill Sewered Effluent," by Isiah Gellman, National Council for Steam Improvement, Portland, Ore.

1:30 p.m. Meetings of Testing Division Service Committees—visitors welcome

3:30 p.m. Meetings of Testing Division Product Committees—visitors welcome

WEDNESDAY, AUG. 19, 1959 (PACIFIC SECTION DAY)

9:00 a.m. Technical Session

5. "Experience with 80-Grit Touch-up Method," by F. Dinger, Puget Sound Pulp & Timber Co., Bellingham, Wash.
6. "A Method for Grinding and Calibration of the Valley Beater," by V. H. Poole, Powell River Corp., Powell River, B. C., Canada.
7. "Effect of Electrolyte in the Valley Beater Test," by B. B. Thomas, Rayonier Inc., Shelton, Wash.
8. "Practical Pulp Strength Evaluation Techniques," by A. A. Mann, S. D. Warren Co., Cumberland Mills, Me.
9. "Use of Commercial-size Refiner for Pulp," by J. L. McAndie, Morden Machines Co., Portland, Ore.

2:00 p.m. Technical Session

Pulp Evaluation Panel:

R. P. Whitney, Moderator, The Institute of Paper Chemistry, Appleton, Wis.
L. S. Mordman, Oy Keskuslaboratorio-Central-laboratorium Ab, Helsingfors, Finland
J. d'A. Clark, Consultant, Victoria, B. C., Canada
W. B. Campbell, Consultant, Montreal, P. Q., Canada
J. A. Van den Akker, The Institute of Paper Chemistry, Appleton, Wis.

Evening

Social hour and banquet
Banquet speaker: Stuart Holbrook. Subject: "Daylight in the Swamp."

THURSDAY, AUG. 20, 1959

Morning

Committee Meetings
Service and Product Committees—Visitors Welcome

2:00 p.m. Technical Session

10. "Interlaboratory Testing of Pentosan Methods," by W. K. Wilson, National Bureau of Standards.
11. "Gas Chromatographic Analysis," by D. F. Adams and R. K. Koppe, State College of Washington, Corvallis, Wash.
12. "Interlaboratory Variations of the Tearing Test," by T. W. Lashof, National Bureau of Standards.
13. "Boxboard Scorability Tester," by R. L. Lewis and C. Eckhart, Ohio Boxboard Co., Rittman, Ohio, and A. T. Luey, Boxboard Research and Development Assoc., Kalamazoo, Mich.
14. Report on the Newsprint Evaluation Program, by the American Newspaper Publishers Assoc., Research Institute, Easton, Pa.
15. "Methods for Using the I.G.T. Printability Tester," by A. Glassman, R. R. Donnelley & Sons Co., Chicago, Ill.

FRIDAY, AUG. 21, 1959

Woods Trip to Crown Zellerbach Clatsop Tree Farm

8:30 a.m. Leave Hotel

6:00 p.m. Return to Hotel

The Tenth Testing Conference begins on Monday, August 17, with two outstanding mill visits. The delegate will have a choice between two large and extensively diversified mills. The Crown Zellerbach mill in Camas, Wash., contains 14 paper machines and makes a large assortment of different papers and boards. Visitors will especially enjoy the trip through their extensive research and development facilities. The Weyerhaeuser mill in Longview, Wash., uses all of the tree in a very extensive wood products plant. One of the high lights of this mill tour will be a trip through the wood products research and development center where you will see products of tomorrow. Families will be welcome and they will enjoy either of these tours. A buffet supper has been planned for Monday evening to give everyone the opportunity to become better acquainted early in the conference.

TAPPI visitors to Crown Zellerbach's Clatsop Tree Farm will see the word's largest Sitka spruce and Douglas-fir during their Friday, August 21 tour. The spruce above, is 195 ft. tall, 15 ft. 9 in. diam. and estimated to be at least 700 years. Its 60,000 plus board feet of lumber would build six two-bedroom frame homes or make upwards of 100 tons of paper

The technical program beings on Tuesday with the opening session devoted to interesting and diversified papers. Tuesday afternoon will be devoted to committee meetings. The committee meetings will be open to all.

Wednesday is Pacific Section Day. This means there will be a large contingent from the Pacific Section, the oldest local section of TAPPI. They are observing their 30th anniversary this year, and are looking forward to the opportunity for this joint meeting with other TAPPI members and friends. Wednesday morning will be devoted to the presentation of papers of general and manifold interest. The high light of the technical sessions is scheduled for Wednesday afternoon. Roy P. Whitney, vice-president and dean, The Institute of Paper Chemistry, will lead a panel of international experts in discussing the practical and theoretical aspects of pulp evaluation. This pulp evaluation panel will be particularly stimulating and educational to technical representatives from all users and producers of pulp. The conference banquet is scheduled for Wednesday evening and the speaker will be Stuart Holbrook. Mr. Holbrook is a very well known author and lecturer and will give an interesting and homespun talk on the history of logging on the North American continent.

Thursday morning will be devoted to committee meetings which are open. The technical program will conclude Thursday afternoon with the presentation of more outstanding papers. The technical program has been very ably put together by S. M. Chapman of The Pulp & Paper Research Institute of Canada.

The theme of the conference is pulp evaluation due to the importance of pulp production in the Pacific Northwest. However, papers will be presented on paper testing also, and all 17 TAPPI testing committees will hold meetings during the conference. There will be valuable ideas for everyone who is able to attend this conference.

Another high light of the conference is the woods trip planned for Friday. This is an opportunity to see logging in one of the large tree farms of the Northwest. Visitors will see many different types of logging including the cutting of some large old growth trees in the Clatsop Tree Farm of the Crown Zellerbach Corp. A real loggers' lunch will be served during the trip through this tree farm.

Fiber Attraction and Bonding Symposium

A TAPPI symposium on the Fundamentals of Fiber Attraction and Bonding will be held at The Institute of Paper Chemistry, Appleton, Wis., Sept. 9–11, 1959.

The program will cover the gamut of forces involved between fibers in dilute suspension, in wet-web state, in dry or semidry condition, and the effect of wet strength and dry strength additives under these various conditions.

The symposium is jointly sponsored by the TAPPI Fundamental Research Committee and the Wet Strength and Interfiber Bonding Committee. Members of the Planning Committee for the symposium are:

General Chairman: Roy P. Whitney and John W. Swanson, The Institute of Paper Chem., Appleton, Wis.
Local Arrangements Chairman: J. J. Shipman, Kimberly-Clark Corp., Neenah, Wis.
Publicity Chairman: C. S. Maxwell, American Cyanamid Co., Stamford, Conn.
Program Chairman: R. N. Griesheimer, The Mead Corp., Chillicothe, Ohio.

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C. S. Maxwell, American Cyanamid Co., Publicity Chairman

Fiber Attraction and Bonding Symposium

Program Committee: K. W. Britt, Scott Paper Co., Chester, Pa.; G. K. Dickeman, Consolidated Water Power and Paper Co., Wisconsin Rapids, Wis.; R. A. A. Hentschel, E. I. du Pont de Nemours & Co., Wilmington, Del.

Because of space limitations attendance at the symposium will be limited. The first 225 applications will be accepted. Application forms may be obtained from R. P. Whitney, The Institute of Paper Chemistry, Appleton, Wis.

The program for the symposium is given below.

WEDNESDAY, SEPT. 9, 1959

8:30 a.m. Registration

9:30 a.m. Session I, Principles of Adhesion

Chairman, Roy P. Whitney, The Institute of Paper Chemistry, Appleton, Wis.

1. "Fundamentals of Adhesion," by Alfred H. Nissan, Rensselaer Polytechnic Institute, Rensselaer, N. Y.
2. "Structural Aspects of Bonding," by J. A. Van den Akker, The Institute of Paper Chemistry, Appleton, Wis.

12:15 p.m. Luncheon

2:00 p.m. Session II, Development of Wet Web Strength

Chairman, R. N. Griesheimer, The Mead Corp., Chillicothe, Ohio

1. "Fiber Agglomeration and Dewatering," by W. L. Ingmanson and S. T. Han, The Institute of Paper Chemistry, Appleton, Wis.
2. "Wet Web Strength," by W. Brecht and H. Erfurt, Institut für Papierfabrikation, Technische Hochschule, Darmstadt, Germany.
3. "Some Aspects of Wet Web Behavior," by A. Robertson, Pulp and Paper Research Institute of Canada, Montreal, Que.

THURSDAY, SEPT. 10, 1959

9:00 a.m. Session III, Fiber Properties Affecting Adhesion

Chairman, G. K. Dickerman, Consolidated Water Power and Paper Co., Wisconsin Rapids, Wis.

1. "Flexibility of Fibers," by S. F. Kurath, The Institute of Paper Chemistry, Appleton, Wis.
2. "Dimensional Stabilization of Paper," by W. E. Cohen, A. J. Stamm, and D. J. Fahey, U. S. Forest Products Laboratory, Madison, Wis.
3. "Fiber Geometry as Related to Adhesion," speaker to be announced.

12:15 p.m. Luncheon

2:00 p.m. Session IV, Development of Bonds

Chairman, R. A. A. Hentschel, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

1. "Mechanism of Bonding," by W. Boyd Campbell, Consultant, Montreal, Que.
2. "Fiber Surface Area and Bonded Area," by J. W. Swanson, The Institute of Paper Chemistry, Appleton, Wis.
3. "Bonding in Synthetic Fiber Papers," by R. A. A. Hentschel, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

FRIDAY, SEPT. 11, 1959

9:00 a.m. Session V, Role of Additives in Bonding

Chairman, K. W. Britt, Scott Paper Co., Chester, Pa.

1. "Polysaccharide Adhesives," by M. L. Cushing, A. E. Staley Manufacturing Co., Decatur, Ill.
2. "Mechanism of Retention of Resin Additives," by E. F. Thode, J. W. Swanson, S. F. Kurath, The Institute of Paper Chemistry, Appleton, Wis.
3. "Elastomeric Binders," by Paul J. McLaughlin, Rohm & Haas Co., Philadelphia, Pa.

12:15 p.m. Official Luncheon

Chairman, R. P. Whitney, The Institute of Paper Chemistry, Appleton, Wis.

2:30 p.m. Session VI, Panel Discussion and Summary

Chairman, J. W. Swanson, The Institute of Paper Chemistry, Appleton, Wis.

Panel to be composed of speakers at previous sessions.

Ninth Corrugated Containers Conference

The Ninth TAPPI Corrugated Containers Conference will be held Sept. 15–17, 1959, at the Jung Hotel, New Orleans, La.

J. H. Rooney, Continental Can Co., New Orleans, La., is general conference chairman. The conference will consist of two technical sessions on Wednesday. R. C. Hutcheson, Union Bag-Camp Paper Co., Savannah, chairman of the Engineering Committee, sponsor of the morning session, will be moderator. The theme of this session is "Engineering Advances Contributing to the Reduction of Scrap."

The session on Wednesday afternoon is sponsored by the Raw Materials Committee, D. C. Shepard, Jr., Menasha Wooden Ware Corp., Menasha, Wis., chairman. The theme of this session is "What Is Required in Corrugated Board to Compete with the Demands of Modern Packaging?" The moderator is G. R. Wilmer, American Box Board Co., Grand Rapids, Mich.

Two plant tours are scheduled for Thursday, Sept. 17, 1959. One is a tour through the Continental Can Co. plant in New Orleans. The other tour is an all-day trip to Bogalusa where the multiplant operation of Gaylord Container Div., Crown Zellerbach Corp., will be seen.

The Steering and Advisory Councils are scheduled for a meeting Tuesday morning, September 15. The Container Testing Committee has scheduled a meeting also on Tuesday morning. The five Corrugated Containers Division Committees: Engineering, Industrial Engineering, Process and Quality Control, Production, and Raw Materials, have scheduled individual meetings on Tuesday afternoon.

The technical program will be published as soon as it has been completed.

"Combination bleaching of mechanical pulp—following peroxide with a hydrosulfite—can give you brightness gains of 15 to 20 points"

Robert Barton, a technical representative for Virginia Smelting Company, discusses the advantages of 2-stage bleaching from the standpoint of costs and results.

Robert Barton, a "Virginia" technical representative

Q Why does 2-stage bleaching give more brightness gains?

A Because the action of peroxide and hydrosulfite on pulp is not the same. Each gains its effect in a different way, and while there is some overlap in their action, there is a very worth-while cumulative effect. In other words, where you can reasonably expect a brightness gain up to 12 points using either bleach alone, the two bleaches used consecutively jump the results to anywhere from 15 to 20 points.

Q What are the advantages of 2-stage bleaching?

A Lower costs and better results—especially if the mill is now using some agent other than hydrosulfite for its single-stage bleaching. The big financial advantage in switching to a combination bleach goes to the pulp mill now using a single stage of peroxide.

Q How does the 2-stage bleach cut costs?

A The principle is to use as little of the more expensive peroxide as possible. The first stage is a bleach with peroxide. Follow this with a hydrosulfite bleach and you have cut the heart out of bleaching costs, at the same time stepping up the quality of the final product. Higher brightness and lower costs are the main reasons why 2-stage bleaching is becoming increasingly popular.

Q Can this process be applied to any kind of pulp with the same results?

A No, the response varies slightly with the wood from which the pulp is made. Good results have been obtained with spruce, poplar and pine groundwood pulps and with a large number of semimechanical pulps made from many species of caustic or sulfite pretreated hardwoods. Mixtures of chemical and mechanical pulps also respond favorably.

Q How can pulp manufacturers find out whether or not the 2-stage bleach will be advantageous to them?

A Simply by calling on the services of "Virginia." We can bring our lab equipment right into a mill, make tests, and demonstrate the possibilities of 2-stage bleaching under practical operating conditions. We will lend the mill any necessary equipment, find a spot in the system where the hydrosulfite stage can be inserted, and help run a mill trial. If the 2-stage process proves desirable, we will stay on the job and help iron out any technical problems that might arise. Under circumstances like these a pulpmaker can easily see where costs are being cut—and examine the improved final product with his own eyes. This is the kind of technical service on which "Virginia" has built its reputation.

• • •

For more information on 2-stage bleaching with "Virginia" zinc or sodium hydrosulfite, write us giving any technical data that may help us evaluate your problem. We will forward technical literature and run laboratory tests provided you furnish us fresh pulp samples. Or, if you say so, we will have a "Virginia" man call and talk over your requirements with you.

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Fourth Deinking Conference

W. Doyle Boggess, Beveridge Paper Co., chairman of the Deinking Committee, has announced plans for the forthcoming Fourth Deinking Conference to be held on Thursday and Friday, Oct. 8 and 9, 1959, at the Roger Smith Hotel, Holyoke, Mass.

John Wendell, Jr., Tileston & Hollingsworth Co., is conference chairman. Harold Corwin of Diamond Gardner Corp., is program chairman and Thomas Allan, Jr., of Virginia Smelting Co., is conference treasurer.

Papers on new developments in wastepaper reclamation will be presented, and a panel discussion of flotation deinking is in the preliminary arrangements stage. Mill visits to deinking plants in the vicinity are scheduled for both afternoon and a meeting of the Deinking Committee Wednesday evening.

Fourteenth Engineering Conference

V. P. Owens, Combustion Engineering, Inc., New York, N. Y., general secretary of the Engineering Division and technical program chairman for the Fourteenth Engineering Conference has announced the technical program for the conference.

The program will consist of 17 sessions during which 45 papers and two panel discussions will be presented. Two committees, Chemical Engineering and Corrosion will each present two separate sessions.

The conference is scheduled for Oct. 12–15, 1959, in the Penn Sheraton Hotel in Pittsburgh, Pa.

The technical program appears at the end of this article.

Varied hospitality functions ranging from social engagements that include a brunch and style show for women to industrial plant visitations comprise an exciting program for representatives and their wives attending the Engineering Conference.

According to the Local Arrangements Committee under the chairmanship of M. H. Fisher of Westinghouse Electric Corp., plans for the business-social program are rapidly nearing completion to make this year's conference in the Penn-Sheraton Hotel the most outstanding in TAPPI history. Upwards of 1000 people are expected to attend the annual technical conference which is being held in Pittsburgh for the first time.

The ladies'-men's hospitality program will officially get under way for the former on Monday morning with a "get-acquainted" coffee hour which will be followed in the afternoon by a plant tour of the H. J. Heinz Company, Pittsburgh's famed home of the "57 Varieties." Other educational tours highlighting major steel and box making operations, an atomic power plant, and a campus tour of the nationally-known 42-story University of Pittsburgh Cathedral of Learning and Heinz Memorial, are among those scheduled during the 4-day engineering conference.

One of the most interesting and informative features of Tuesday's program is the appearance of Countess Pulaski who will relate personal experiences in her talk on "My Life as a Spy" at the banquet for representatives and their wives that evening.

TAPPI representatives, and the many thousands of visitors expected to tour Pittsburgh during the city's observance of its Bicentennial year, will find significantly appropriate Charles Dickens' vivid description of Pittsburgh in 1842 as "Hell with the lid lifted." While the city's air is now virtually smokeless, scenes inside some of the Pittsburgh mills still fit Dickens' colorful description.

One of two of nature's forces—smoke—which had posed a problem for many years in Pittsburgh history, but which now has been solved, is a dramatic end result of the city's renaissance during the past decade. With the city's conquest over smoke, which came after years of research, stringent city and county abatement ordinances, and industry-wide cooperation, total smoke has been reduced by 90%, and heavy black smoke by 98%, thus giving Pittsburghers and visitors a "new look at the sun." This accomplishment over smoke and Pittsburgh's other nemesis—floods—had sparked a new spirit of civic enterprise that eventually spearheaded the city's $2-billion redevelopment program of how the New Pittsburgh has become the Renaissance City of America and stirred imaginations throughout the world.

Pittsburgh's origin began at its famed point—The Golden Triangle—made possible by the confluence of the Monongahela and Allegheny Rivers to form the Ohio River in 1758. And, it is significant that the history of the New Pittsburgh also began here in 1950. The commonwealth reserved and began developing 36 acres into Point State Park built around the historical Fort Pitt Blockhouse, last remnant of the old redoubt built by the British in 1764.

Altogether, more than 50 new buildings have dotted the Golden Triangle skyline since the renaissance began, and more than 5300 off-street parking spaces have been created since World War II. The state has presently under construction a $140-million, 21-mile nontoll Penn-Lincoln expressway that speeds traffic nonstop East and West.

TAPPI representatives from the East motoring across the Pennsylvania Turnpike to the Engineering Conference site will find the Penn-Lincoln Parkway readily accessible to significant downtown sightseeing attractions. The Parkway ends in the West at an interchange that leads to the $33-million Greater Pittsburgh Airport. Only 20 min. from downtown Pittsburgh, the airport lures as many as 75,000 visitors on a summer Sunday and more than 2 million persons use its facilities annually. The Terminal Building at Greater Pittsburgh Airport—longer than three football fields and seven stories high—is a monument to vision in an age of jets and improved air travel facilities.

While the construction of modern transportation facilities and new office skyscrapers has been an important phase of Pittsburgh's renaissance, it by no means tells the entire story. Of primary consideration has been the elimination of hundreds of acres of slums and blighted areas. Under a broad, imaginative program now nearing completion, the Urban Redevelopment Authority of Pittsburgh has cleared and demolished the 95-acre tract on the eastern end of the Golden Triangle which will make possible a new pattern of streets, a crosstown boulevard and a combination civic auditorium-sports arena with a unique retractable roof. Many other slum areas throughout the Pittsburgh area have also been demolished for new projects, and merchants have spent millions on modernization programs and cleaning their buildings of old grime.

Correlated to these improvements has been an influx of many new industries which have provided additional jobs in the Pittsburgh area. Keeping pace with the new arrivals, and vitally as important is the fact the city's industries have launched expansions and renovations totaling more than a billion dollars in the last decade.

Research, education, and cultural interests have also made tremendous progress through beneficial grants which have helped nurture Pittsburgh's renaissance. Today, Pittsburgh with no less than 50 major research centers, ranks as one of the world's great industrial research capitals. Through sizable gifts ranging in the millions of dollars, the city's institutions of higher learning—University of Pittsburgh, Carnegie Institute of Technology, and Duquesne University—have undertaken expansion programs, thereby increasing the educational opportunities of Pittsburgh's younger generation on whom the city will depend if it is to continued to prosper in the future.

Pittsburgh's rebirth during the past two decades cannot be attributed to a single individual, nor any single organization. It has been the concerted effort of numerous organizations such as the Urban Redevelopment Authority, the

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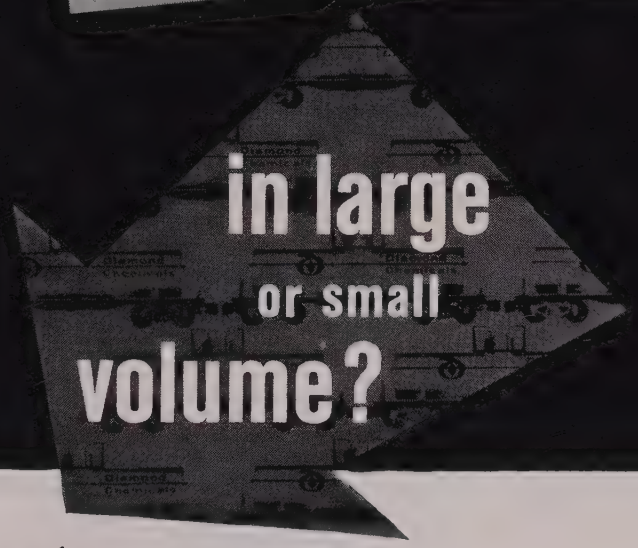

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Allegheny Conference on Community Development, the Regional Planning Association, the Chamber of Commerce and many others. The civic-minded giants of Pittsburgh industry, business, government, commerce, education, culture and thousands of others—their tireless sacrifices, coordinative, and creative efforts have reestablished what was once a slowly-decadent metropolis into a thriving revitalized American city, within whose borders are headquarters of 100 major corporations, the heart of throbbing, basic, heavy industries and home of hundreds of small businesses. These are the characteristics that have again reaffirmed Pittsburgh's proud claim as the "Workshop of the World."

Fourteenth Engineering Conference

TENTATIVE PROGRAM

Oct. 11–15, 1959
Hotel Penn Sheraton
Pittsburgh, Pa.

SUNDAY, OCT. 11, 1959

10:00 a.m.–12:00 Noon Staff Advisory Committee (Parlor B)
1:00 p.m.–5:00 p.m. Committee Consultations (Parlor B)
4:00 p.m.–8:00 p.m. Registration (Fort Duquesne Room)

MONDAY, OCT. 12, 1959

8:00 a.m.–5:00 p.m. Registration (Fort Duquesne Room)

9:00 a.m. First Session, General (Urban Room)

Opening remarks, J. R. Curtis, Scott Paper Co., *General Chairman*

9:15 a.m.–12:00 Noon Second Session (Urban Room)

Steam and Power, R. V. Knapp, Mosinee Paper Co., Mosinee, Wis., *Chairman*

1. "Progress Report on Atomic Power," by J. W. Magee, Jr., and J. H. Wright, Westinghouse Electric Corp., Phila., Pa.
2. "Procedures for Allocating Costs of Process Steam and Electric Power in Paper Mill Power Plants," by G. D. Farrar, General Electric Co., Atlanta, Ga.

Panel Discussion: Economic Aspects of Pressures and Temperatures for Pulp and Paper Mills. Moderator: J. G. Hoad, John G. Hoad & Assoc., Ypsilanti, Mich.

1. "Turbines-Generators," by I. H. Landes, General Electric Co., Schenectady, N. Y.
2. "Power Boilers," by H. B. Wallace, Jr., Foster Wheeler Corp., New York, N. Y.
3. "Recovery Boilers," by W. J. Darmstadt, Babcock and Wilcox Co., Barberton, Ohio, and F. W. Hochmuth, Combustion Engineering, Inc., New York, N. Y.
4. "Mill Experience," by T. J. Judge, International Paper Co., Mobile, Ala.

9:15 a.m.–12:00 Noon Third Session (Monongahela Room)

Maintenance Engineering, R. Q. Conner, The Crossett Co., Crossett, Ark., *Chairman*

1. "The New Challenge in Tomorrow's Maintenance," by H. P. Evans, C. F. Braun & Co., Alhambra, Calif.
2. "Up-to-Date Training in Paper Mills," by H. W. Smith, St. Regis Paper Co., Pensacola, Fla.
3. "Organized Lubrication—A Case Report," by J. Box, Gaylord Container Corp., Division of Crown Zellerbach Corp., Bogalusa, La.

12:30 p.m.–1:45 p.m. Committee Luncheons
Civil Engineering (Grant Room)
Drying (Sky Room)
Engineering Economics (Club Room)
Industrial Engineering and Materials Handling (Park View Room)
Maintenance Engineering (Room 566)
Sanitary Engineering (Room 766)

2:00 p.m.–5:00 p.m. Fourth Session (Urban Room)

Electrical Engineering, R. F. Sorenson, St. Regis Paper Co., Jacksonville, Fla., *Chairman*

1. "Electrical Control of Fourdrinier Helper Drives," by S. J. Campbell, Westinghouse, Pittsburgh, Pa., and D. C. Pier, Beloit Iron Works, Beloit, Wis.
2. "Semiconductor Rectifier Power Supplies in the Paper Industry," by G. E. Shaad, General Electric Co., Schenectady, N. Y.
3. "Safety in Mill Electrical Work," by M. L. Perkins, Fraser Paper Co., Madawaska, Me.
4. "Power Transfer Within Wet Felts of Paper Machines," by M. J. Osborne, Bowaters Southern Paper Corp., Calhoun, Tenn., and I. Peters, F. C. Huyck Co., Rensselaer, N. Y.

2:00 p.m.–5:00 p.m. Fifth Session (Monongahela Room)

Mechanical Engineering, M. N. Davis, Kimberly-Clark Corp., Neenah, Wis., *Chairman*

1. "Recent Improvements in Wood Grinders for Insulating Fiberboard Manufacture," by W. F. Fields, Armstrong Cork Co., Lancaster, Pa.
2. "Extensible Paper Machinery," by C. E. Hill, West Virginia Pulp and Paper Co., Covington, Va.
3. "A Factual Appraisal of the Horizontal Size Press from the Construction and Design Aspect," by H. A. Smith, Rice Barton Corp., Worcester, Mass.

5:30 p.m. and 7:00 p.m. Sixth Session (Urban Room)

Mill Tours Committee
Film "Atomic Power at Shippingport"

6:00 p.m. Process Instrumentation Committee (Parlor B)

TUESDAY, OCT. 13, 1959

8:00 a.m.–5:00 p.m. Registration (Duquesne Room)

9:00 a.m.–12:00 Noon Seventh Session (Urban Room)

Civil Engineering, W. Pittam, Stone and Webster Engineering, Inc., Boston, Mass., *Chairman*

1. "Tanks and Pressure Vessels for the Pulp and Paper Industry," by R. A. Jackson, Chicago Bridge & Iron Co., Seattle, Wash.
2. "Allowable Wind Loads on Various Structures," by J. H. Bringhurst, J. E. Sirrine Co., Greenville, S. C.
3. "Various Types of Outfall Lines into Bodies of Water," by W. Pittam, Stone & Webster Engineering Co., Boston, Mass.

9:00 a.m.–12:00 Noon Eighth Session (Allegheny Room)

Engineering Economics, G. W. Sargent, Central Fibre Products Co., Quincy, Ill., *Chairman*

1. "The Dollars and Sense of Capital Equipment Justification," by W. Bricker, Diamond-Gardner Board and Carton Co., Middletown, Ohio.
2. "Calculating the Return on Proposed Projects," by R. I. Reul, Food Machinery & Chemical Corp., New York, N. Y.

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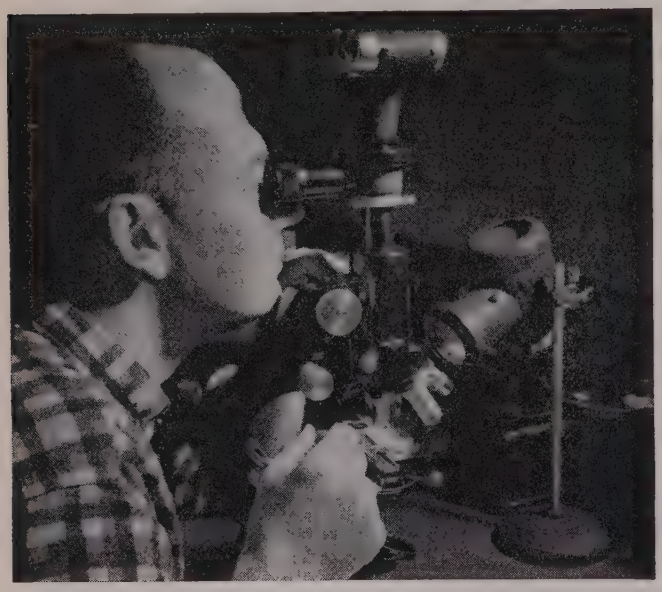
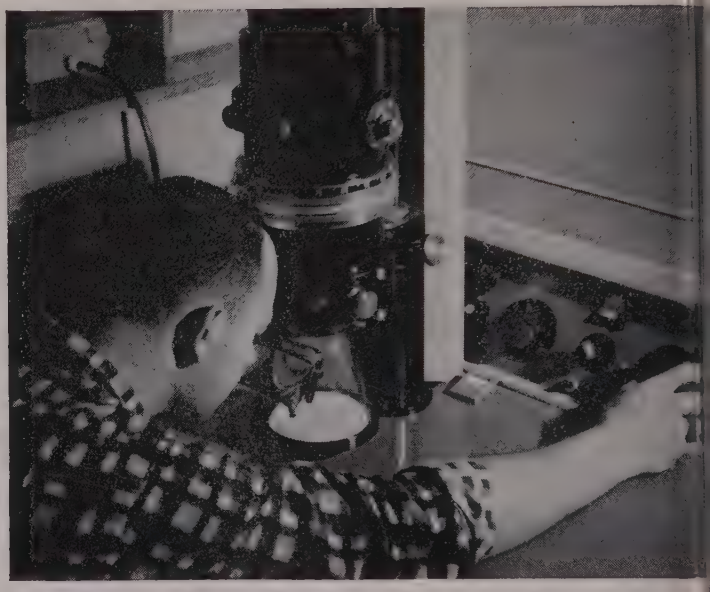
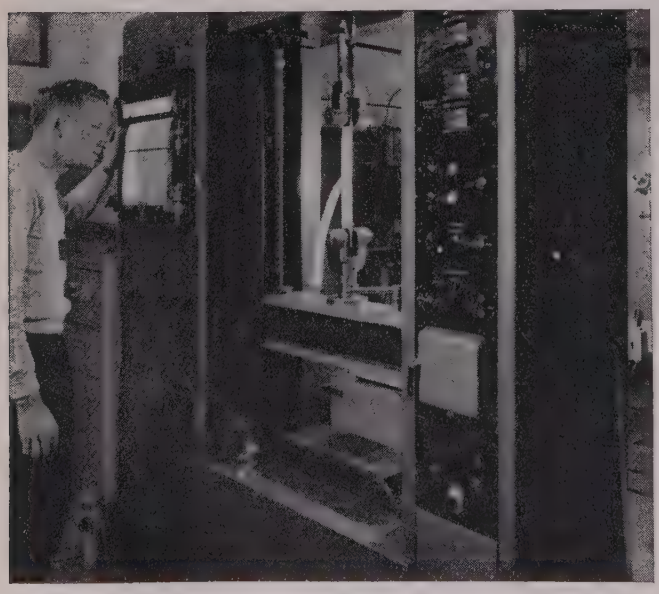
1. Koppers new Kobuta Development Laboratory.



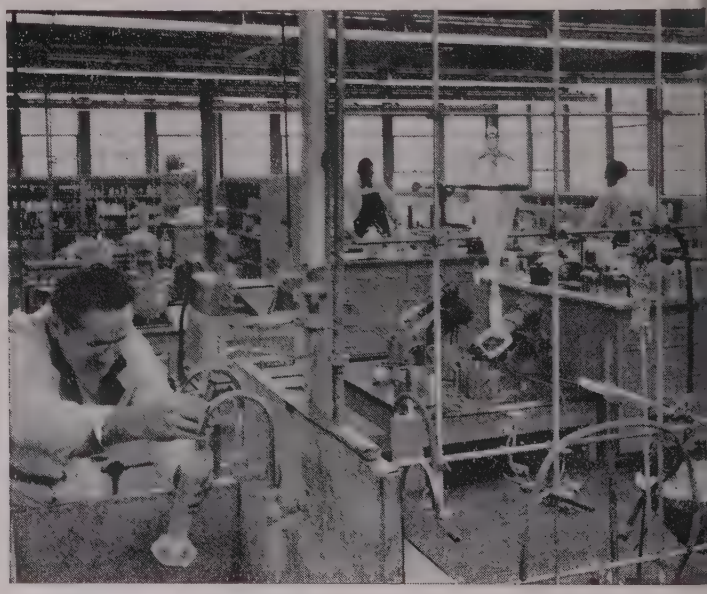
2. Here, coatings made from DYLEX K-52 and a conventional latex take photo comparison test.



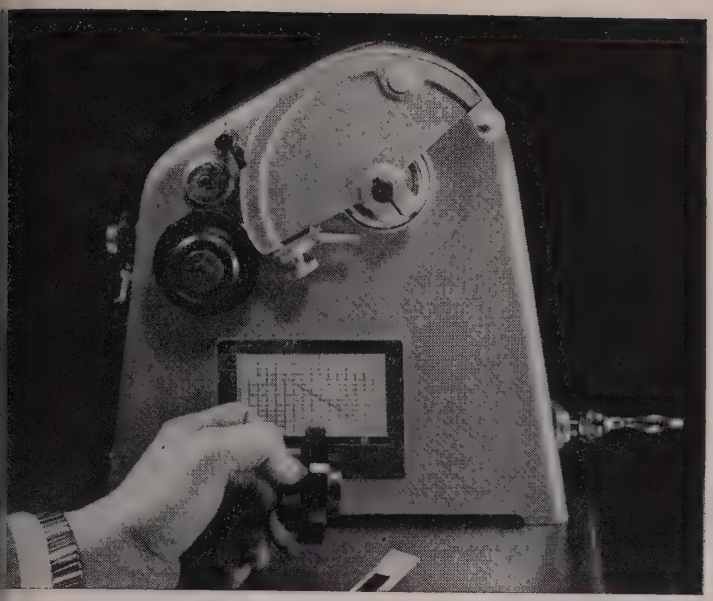
3. Latices are first checked for particle size in the Electron Microscope. Uniformity is essential for fine coatings.



4. The Instron Tester—this instrument measures tensile and flexural strength of coated paper, or films for coated paper.

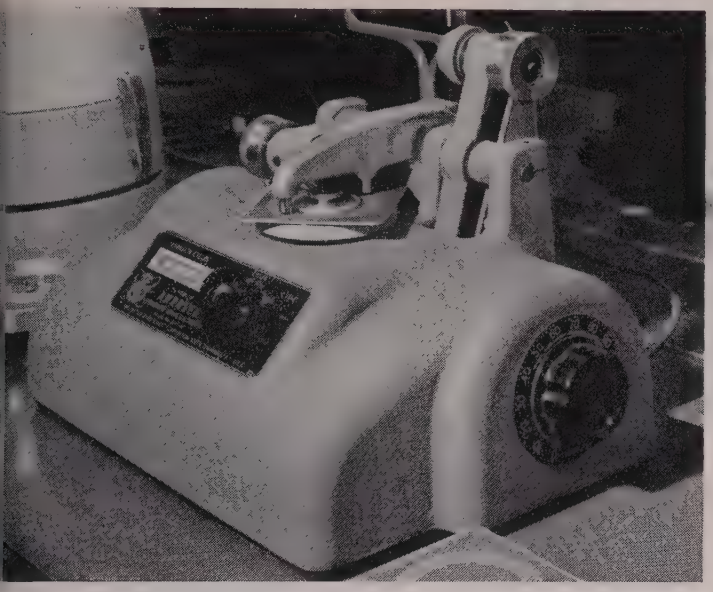


5. When the coating passes these tests, it's ready for the Paper Applications Laboratory.



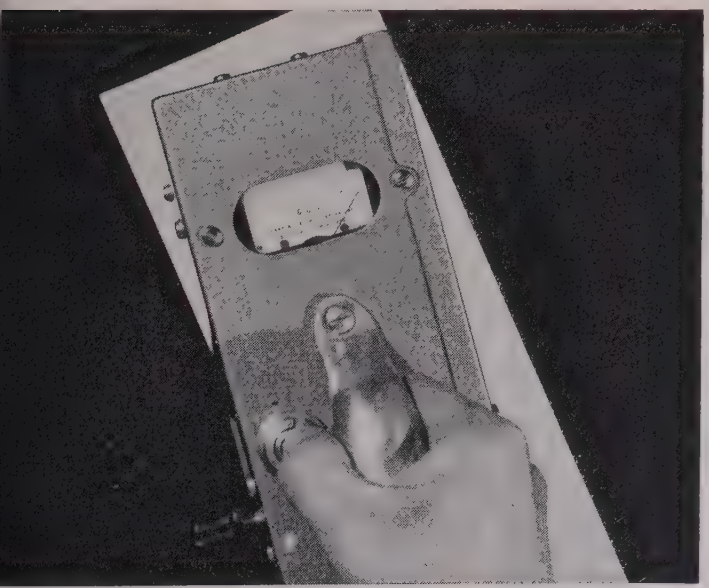
6. The IGT pick test determines the adhesive strength of the coating.

7. The coating is applied to paper in a new experimental paper coater. This machine **actually duplicates** mill conditions.



8. The Tabor Abrasor determines the wet rub resistance of the coating.

9. After the sheets have been calendered and tested for pick resistance, they are checked for smoothness.



10. Here, quality coated paper is carefully checked for gloss.

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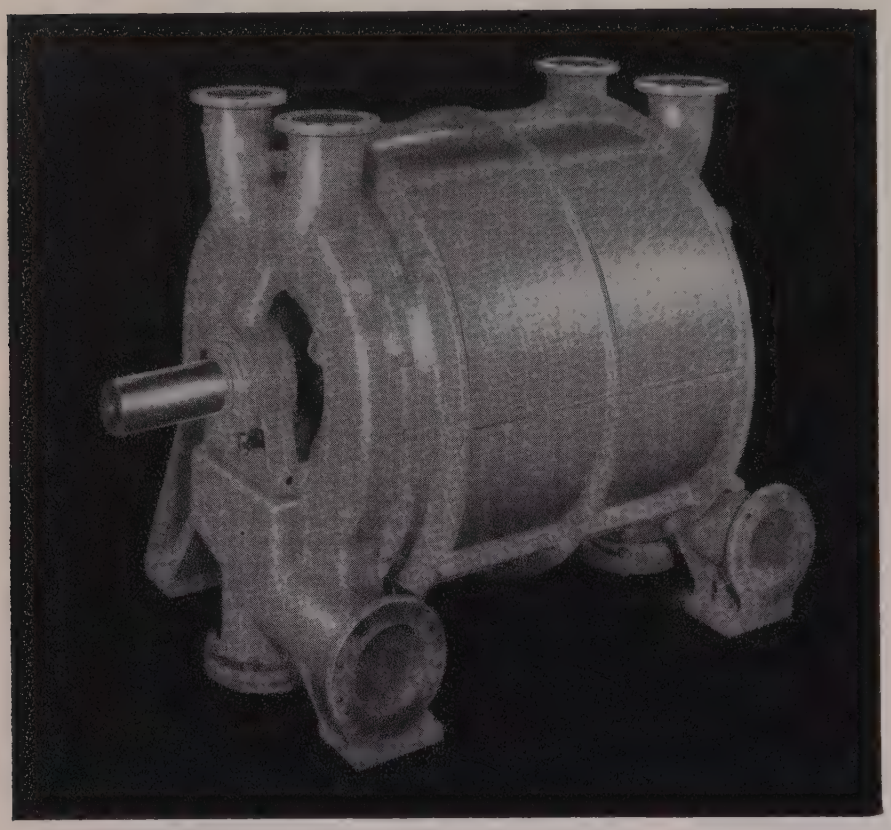
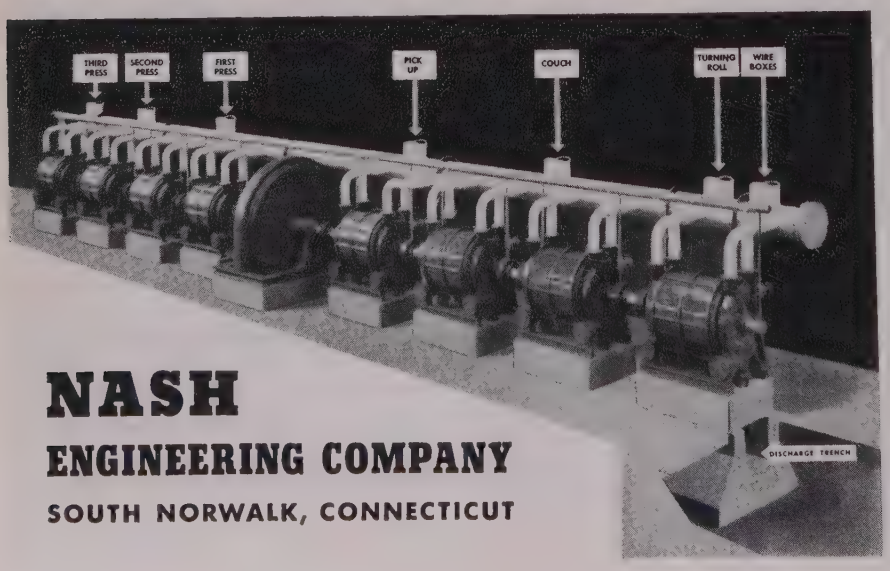


The Nash 5308-A has four separate suction inlets, each of which functions independently of the others. This offers the machine operator great flexibility, since these may be used in any desired combination to produce a variety of capacities and vacuums.

Picture below shows simple drive possible and flexibility of piping permitting capacities to exactly fit requirements of different sections of the paper machine.



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9:00 a.m.–12:00 Noon. Ninth Session (Monongahela Room)

Process Instrumentation, J. K. POWELL, Bowaters Southern Paper Corp., Calhoun, Tenn., *Chairman*

1. "Experimental Recovery Unit" (speaker not confirmed).
2. "A Versatile New Consistency Regulator," by K. I. Mumme and N. W. Swanson, Kimberly-Clark Corp., Neenah, Wis.
3. "How One Mill Handles Instrumentation (Engineering, Specifications, Applications, Maintenance)" speaker not confirmed.

12:30 p.m.–1:45 p.m. Committee Luncheons
Chemical Engineering (Room 766)
Corrosion (Aero Room)
Electrical Engineering (Grant Room)
Fluid Mechanics (Room 566)
Mechanical Engineering (Park View Room)
Steam and Power (Sky Room)

2:00 p.m.–5:00 p.m. Tenth Session (Urban Room)

Fluid Mechanics, N. SHOUMATOFF, West Virginia Pulp & Paper Co., New York, N. Y., *Chairman*

1. "Patterns of Circumferential Flow in Cylindrical Annuli," by A. H. Nissan and F. C. Haas, Rensselaer Polytechnic Inst., Troy, N. Y.
2. "Flow Instability on the Fourdrinier Wire-Progress Report of TAPPI Project No. 156," by C. S. Yih and A. C. Spengos, Univ. of Michigan, Ann Arbor, Mich.
3. "Paper Machine Inlet Performance with Relation to the Fourdrinier Wire," by J. C. Nelson, Kimberly-Clark Corp., Neenah, Wis.
4. "The Effects of Fibers on Velocity Distribution, Turbulence and Flow Resistance of Dilute Suspensions—Report of TAPPI Project No. 147," by J. W. Daily and G. Bugliarello, Massachusetts Institute of Technology, Cambridge, Mass.

9:15 a.m.–12:00 Noon Eleventh Session (Monongahela Room)

Sanitary Engineering, W. A. MOGGIO, Lake States Yeast Corp., Rhinelander, Wis., *Chairman*

Theme: "Air—Its Role in Waste Treatment and Disposal"

1. "Flotation Methods," by L. L. Klinger, Central States Engineering, Inc., Appleton, Wis.
2. "Oxidation Lagoons," speaker from National Council for Stream Improvement, New York, N. Y.
3. "River Reaeration," speaker from Sulphite Pulp Mfgr's. Research League, Appleton, Wis.

6:00 p.m.–7:00 p.m. Cocktail Party (Urban Room)

7:00 p.m. Banquet (Ball Room)

Toastmaster, M. H. Fisher, Westinghouse Electric Co., East Pittsburgh, Pa.

Conference Arrangements Chairman
Speaker: (to be announced later)

10:00 p.m.–1:00 a.m. Dance (Ball Room)

WEDNESDAY, OCT. 14, 1959

7:30 a.m. Chemical Engineering Author's Breakfast (Parlor B)

8:00 a.m.–12:00 Noon Registration (Duquesne Room)

9:00 a.m.–12:00 Noon Twelfth Session (Monongahela Room)

Corrosion, E. W. Hopper, Pittsburgh, Pa., *Chairman*

1. "The Use of Titanium in the Pulp and Paper Industry," by W. K. Boyd, Battelle Memorial Institute, Columbus, Ohio.
2. "Corrosion of Fourdrinier Wires," by J. Gerhauser, Appleton Wire Works, Appleton, Wis.
3. "Digester Corrosion at Union Bag-Camp Paper Corp.," by H. B. Harris, Jr., and L. Parks, Union Bag-Camp Paper Corp., Savannah, Ga.
4. "Corrosion of Mild Steel in Alkaline Pulping Liquors. Final Report of Project Partially Sponsored by TAPPI Research Grant No. 86," R. B. Kesler, The Institute of Paper Chemistry, Appleton, Wis.

9:00 a.m.–12:00 Noon Thirteenth Session (Urban Room)

Industrial Engineering and Materials Handling, by B. C. Kendall, Crown-Zellerbach Corp., San Francisco, Calif., *Chairman*

1. "Do You Really Mean It?" by J. E. Angle, U. S. Steel Corp., Pittsburgh, Pa.
2. "Broke Handling at Glatfelter's," by R. D. Owen, P. H. Glatfelter Co., Spring Grove, Pa.

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9:00 a.m.–12:00 Noon Fourteenth Session (Allegheny Room)

Chemical Engineering, I, Systems Engineering and Operations Research, E. F. Thode, The Institute of Paper Chemistry, Appleton, Wis., *Chairman*

1. "Progress Understanding Required for Systems Engineering," by J. O. Hougen, Monsanto Chemical Co., St. Louis, Mo.
2. "Computer Control in the Pulp and Paper Industry," by T. M. Stout, Thompson-Remo-Wooldridge Products Co., Los Angeles, Calif.
3. "Analog Simulation of a Complex Level Control System," by K. I. Mumme, Kimberly-Clark Corp., Neenah, Wis.
4. "The Application of a Unique Analog Computer to Product Distribution," by C. E. McCarty, Scott Paper Co., Chester, Pa.

12:30 p.m. Engineering Division Luncheon (Ball Room)

J. R. Curtis, Scott Paper Co., Chester, Pa., *Division, Chairman*

Speaker: (to be announced later)

2:00 p.m.–5:00 p.m. Fifteenth Session (Urban Room)

Chemical Engineering, II, A. W. Plummer, Hudson Pulp and Paper Co., So. Windham, Me., *Chairman*

1. "Post Pressure Roll Yankee Drier Sheet Consistency Determination by Material and Enthalpy Balance," by W. A. Dickens, Kimberly-Clark Corp., Neenah, Wis.
2. "Wetting Agent Influence on Water in Sheets After Free Drainage," by R. V. Touchette, International Paper Co., Ticonderoga, N. Y., and L. C. Jenness, University of Maine, Orono, Me.
3. "Operation of Continuous Cold Caustic Pulping Systems," by J. Dyer, Bowaters Southern Paper Corp., Calhoun, Tenn.
4. "Thermodynamic Properties of the System Na_2SO_3-$NaHSO_3$," by R. S. Morgan, Mohawk Paper Mills, Inc., Conoes, N. Y.

2:00 p.m.–5:00 p.m. Sixteenth Session (Monongahela Room)

Drying, S. G. Holt, Consolidated Water Power and Paper Co., Wisconsin Rapids, Wis., *Chairman*

1. "The New Airborne Flakt Drier," by G. E. Jackson, Bowaters Southern Paper Corp., Catawba, S. C.
2. "Static Pressing of Wet Sheets and Felts," R. E. White, Villanova University, Villanova, Pa.
3. "Survey of Nondestructive Drying Methods," by D. M. McCutcheon, Westinghouse Atomic Power Div., Pittsburgh, Pa.
4. "Progress Report on Project 505—Drying Rates of High Pressure Driers," by L. G. Janett, J. O. Ross Engineering Corp., Chicago, Ill.

2:00 p.m.–5:00 p.m. Seventeenth Session (Allegheny Room)

Corrosion, II, E. W. Hopper, Pittsburgh, Pa., *Chairman*

"Overlay Repair of Alkaline Pulping Digesters"
Moderator: E. W. Hopper
Panel: Z. Blanchard, Portland Co., Portland, Me.; H. M. Canavan, Mutual Boiler and Machinery Ins. Co., Waltham, Mass.; E. G. Holmberg, International Nickel Co., Inc., New York, N. Y.; D. Innes, International Paper Co., So. Kraft Division, Mobile, Ala.; W. H. Parker, Hartford Steam Boiler and Ins. Co., Hartford, Conn.; P. Zouck, Alloy Cladding Co., Baltimore, Md.

4:30 p.m.–5:30 p.m. Engineering Data Committee (Park View Room)

6:00 p.m.–8:00 p.m. Division Staff Dinner (Grant Room)

THURSDAY, OCT. 15, 1959

9:15 a.m. Mill Tour A—Jessop Steel Co., Washington, Pa.; National Annealing Box, Washington, Pa.

9:15 a.m. Mill Tour B—Westinghouse Test Nuclear Reactor Site, Waltz Mills, Pa.; Westinghouse Research Lab., Pittsburgh, Pa.

Ladies Headquarters—Sunday—Thursday Room 1690

Conference Headquarters—Sunday—Friday Parlor G

13th Alkaline Pulping Conference

The 13th Alkaline Pulping Conference will be held in Jacksonville, Fla., Nov. 4–6, 1959, at the new Robert Meyer Hotel.

J. McK. Limerick, Bathurst Power and Paper Co., Montreal, Canada, chairman of the Alkaline Pulping Committee, is also general conference chairman. S. P. Kaptain, St. Regis Paper Co., Jacksonville, Fla., is chairman of the Local Arrangements Committee. Assisting Mr. Kaptain are R. E. Phinney, Container Corp. of America, Fernandina, Fla., in charge of hospitality; R. A. Flick, Brunswick Pulp and Paper Co., Brunswick, Ga., in charge of printing; W. W. Stuart, Hudson Pulp and Paper Co., Palatka, Fla., in charge of plant tours; W. B. Hobbs, Jr., and G. R. Miller, both of Owens Illinois Glass Co., Jacksonville, Fla., in charge of hotel arrangements and registration; and B. L. Rauschert, Rayonier, Inc., Fernandina Beach, Fla., in charge of publicity for the conference.

As in the past several years, three of the four technical sessions will be sponsored by the Alkaline Pulping Committee. K. D. Running, Halifax Paper Co., Roanoke Rapids, N. C., is technical program chairman for these three sessions.

The fourth technical session is sponsored by the Semichemical Pulping Committee, P. E. Trout, Waldorf Paper Products Co., St. Paul, Minn., chairman. R. E. Collins, The Crossett Co., Crossett, Ark., is technical program chairman for the fourth session.

Messrs. Running and Collins have in preparation technical programs which appear to be of significant interest to the industry. The programs will be announced as soon as they are completed.

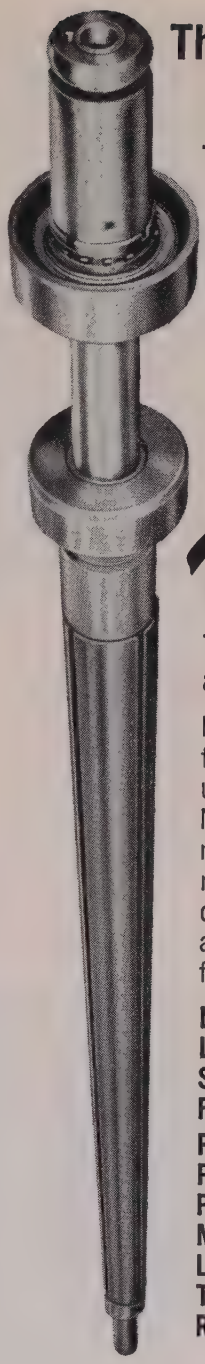
Tenth Coating Conference

The Tenth Coating Conference of the Technical Association of the Pulp and Paper Industry was held at the Statler-Hilton Hotel, Boston, Mass., May 25–27, 1959. Attendance by 801 registrants made this by far the largest Coating Conference to date. About 30 registrants were from foreign countries, including Canada, Italy, England, West Germany, Sweden, Mexico, Brazil, Japan, and Australia.

The conference included four technical sessions, during which 16 papers were presented, a panel symposium on "Coatings, Past, Present and Future," the annual luncheon, and plant visits. In the account given below, brief résumés of the papers are presented. The full text of all the papers will appear in future issues of *Tappi*.

The TAPPI Coating Conference is sponsored by the Coating Committee of the Coating and Graphic Arts Division. The names of the officers of the sponsoring group and of the conference committee are given below:

R. T. Trelfa, general chairman, Coating and Graphic Arts Division, Perkins-Goodwin Corp.
C. A. Morton, secretary, Coating and Graphic Arts Division, Kimberly-Clark Corp.



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J. T. Loomer, chairman, Coating Committee, Continental Can Co., Inc.
F. Kaulakis, general conference chairman, Consolidated Water Power & Co.
R. W. Hagemeyer, Wyandotte Chemicals Corp.
R. E. Kilty, A. E. Staley Manufacturing Co.
H. R. Hall, Riegel Paper Corp., Acme, N. C.
F. R. Marchetti, Titanium Pigment Corp.

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C. W. Cairns, Dow Chemical Co.
H. J. Connell, S. D. Warren Co.
L. A. Gilbertson, Marathon, a Division of American Can Co.
J. T. Goodwin, Corn Ind. Research Foundation, Inc.
R. W. Martinek, Kimberly-Clark Corp.
A. W. Neubauer, Crown Zellerbach Corp.
P. W. Plante, St. Regis Paper Co.
J. C. Tongren, Watervliet Paper Co.
W. E. Welliver, Jr., Minerals & Chemicals Corp. of America
C. G. Whelpton, Champion Paper & Fibre Co.

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H. L. Mellen, vice-chairman, registration, Hercules Powder Co.
W. J. Wood, Jr., registration, Allied Chemical Corp.
E. B. Norman, registration, B. F. Perkins & Co.
J. G. Bullard, registration, Corn Products Refining Co.
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H. N. Lowell, meeting room arrangements, Dewey & Almy Chemical Co.
M. Stiles, meeting room arrangements, Time, Inc.
J. F. Walsh, meeting room arrangements, A. E. Staley Manufacturing Co.

TECHNICAL SESSIONS

First Session

The first technical session of the conference was called to order at 10:30 a.m., Monday, May 25th. After opening remarks by General Chairman F. Kaulakis, J. T. Loomer, Coating Committee chairman, introduced F. H. Frost, S. D. Warren Co., Cumberland Mills, Me., who presented a eulogy of Edwin Sutermeister, to whom the 10th Coating Conference was dedicated. The text of Dr. Frost's address follows:

Eulogy to Edwin Sutermeister

This, the 10th Annual Coating Conference, is dedicated to the memory of Edwin Sutermeister, one of the first of the scientific pioneers of our industry.

Dr. Sutermeister ("Sutie" to all of us who knew him) was known world-wide wherever paper was made or used, chiefly because of the books he wrote. He was known as a scientist of impregnable integrity, as a teacher, and as an originator of important processes and products. His work speaks for the man he was and as an author his friends were legion.

As a person, to those of us who knew him for many pleasurable years, Sutie was individualistic, forthright, helpful, sympathetic, and frequently both blunt and frank.

Sutie's bluntness was one of the most interesting facets of his character. Sutie was blunt because he feared being known as an overly sympathetic individual. He was generous and lent many a helping hand to those in need of help but always anonymously. When he was pleased with something you had accomplished his compliments were brief, and when he himself was complimented, you'd never hear of it from him.

Sutie as a man was refreshing to know, patient with young people willing to work hard—patient with the stubborness with which nature withholds its secrets. He was careful, painstaking, slow, practically never wrong, yet these characteristics were combined with a real originality. It took originality, for example, to develop a starch adhesive for coated papers in 1903 and to invent the chlorination of starch to make it possible.

Sutie, during the last few weeks of his life exhibited mankind's brightest flame—courage. Courage to face the inevitable with quiet good humor. Possibly a little impatient to get the matter settled and finished—possibly a little impatient to discover the next step after mortal life.

Courage, curiosity, faith, a little impatience—all the hallmarks of a true scientist.

Technical Papers. P. W. Plante, St. Regis Paper Co., Deferiet,

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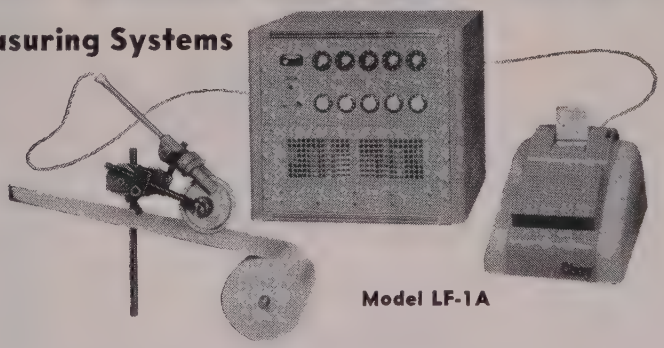

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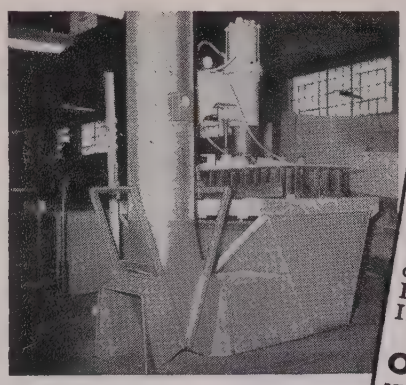
Tenth Coating Conference Luncheon, Statler-Hilton Hotel, Boston, Mass., May 27, 1959

N. Y., was chairman of the technical portion of the first session.

R. W. Hagemeyer, Wyandotte Chemical Co., Wyandotte, Mich., presented "Progress Report No. 2, TAPPI Project 600, The Effect of Pigment Combinations and Coating Solids on Particle Packing and Sheet Characteristics." Other authors of this paper were: J. V. Robinson, Mead Corp., Chillicothe, Ohio; C. E. Kilpatrick, S. D. Warren Co., Cumberland Mills, Me.; H. M. Hughson, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis. Mr. Hagemeyer reported that particle packing had been determined for water dispersions of three basic shapes, the plate, the needle, and the rhomb, both alone and in combinations. The objective was to show how important properties of the pigment suspension depend upon particle packing. Particle packing was expressed in terms of relative sediment volume (RSV). High solids mixing in the presence of chemical dispersants was used to provide proper dispersion. The findings were as follows: First, RSV is dependent upon shape or combination of shapes. Second, RSV correlates quite well with viscosity as measured at both high and low rates of shear; high RSV giving high viscosity, low RSV giving low

Automatic disposal of large broke sheets accomplished with use of crusher-shredder. Shredded pieces flow easily thru system, and are ready for proper baling or reprocessing.



Automatic trim removal from box stripper also provides a floor level pick up to keep the entire area clear.

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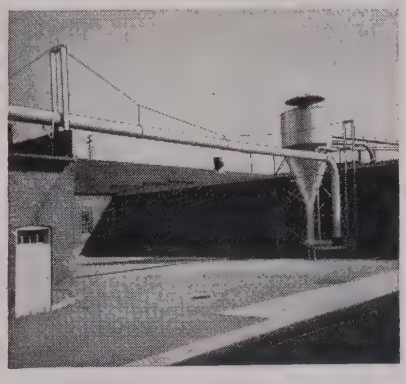
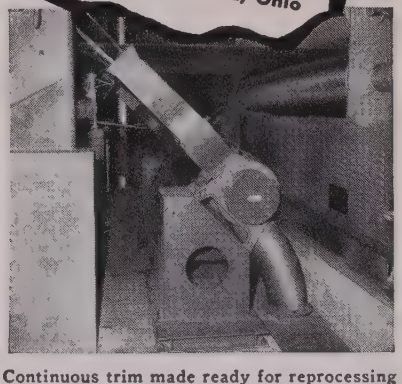


Catenary suspension used to keep yard clear. Pipe runs from transfer collector to separate reprocessing location. Twenty feet of head room allowed for trucks and equipment.



Continuous trim made ready for reprocessing or baling with automatic cutter which is designed as an integral part of the pneumatic system. Cutter accepts continuous lengths of solid fibre to 3/16" thickness, chipboard to 1/4" thickness, widths to 7".

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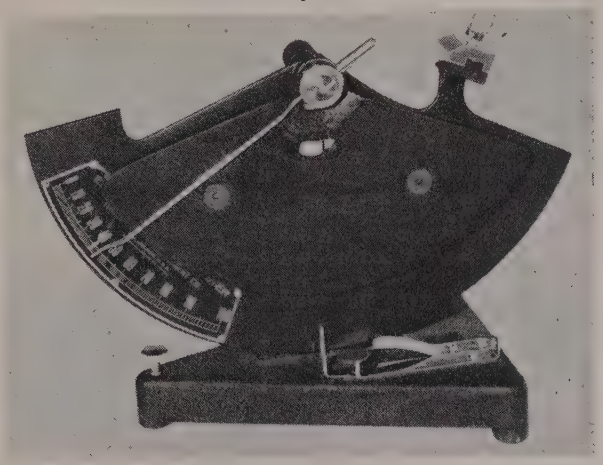

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viscosity. Third, RSV increases with dilution because of flocculation which takes place. Further the plot of RSV versus particle shape or combination of shapes is similar in appearance at all dilutions. Fourth, RSV as measured by centrifuging with water present, or by filtration with water removal, gives similar results. Fifth, while the RSV does not give a good indication of drainage rate, some correlation may exist. The areas for further investigation in the continuation of this project are outlined.

The Interpretation of Statistical Data Obtained in the Coating Process was the title of the second paper, by R. C. Chase, University of Maine, Orono, Me. Mr. Chase stated that some of the variables in the coating process had been analyzed statistically using multiple regression analysis techniques. Eighty-seven different observation equations were used in the analysis with the following independent variables: particle size of clay, amount of dispersing agent, amount of adhesive, pH coating weight, per cent solids of color, machine operating speed, and Brookfield viscosity of 20, 50, and 100 r.p.m. The dependent variables used were Dennison wax pick, Sheffield smoothness, IGT printability, Vandercook printability, and Clayborne printability. The results indicate that several of these variables are significant at the 1% confidence level.

Second Session

On Monday afternoon, May 25, the second technical session was opened at 2:00 p.m. under the chairmanship of L. A. Gilbertson, Marathon Div., American Can Co., Menasha, Wis.

"The Kohler Coating Method," first public disclosure of this new technique, was presented by J. R. Gunning, Abitibi Power & Paper Co., Georgetown, Ont. Mr. Gunning's co-author was J. B. Kohler, consultant, Woodstock, Ill. Mr. Gunning described details of construction, theory, and use

Conference Committee: F. R. Marchetti, Titanium Pigment Corp.; R. E. Kilty, A. E. Staley Mfg. Co.; F. Kaulakis, Consolidated Water Power & Paper Co.; R. W. Hagemeyer, Wyandotte Chemicals Corp.; and G. E. Hall, Wyandotte Chemicals Corp.

of the Kohler coating method. In this method, the paper contacts the coating color in a pond, and is smoothed and metered by a rigid blade which comprises the leaving boundary of the pond. The metering action is controlled by an air jet impinging on the back of the web, in juxtaposition to the rigid blade.

The apparatus is arranged so that the web is not deflected by either the blade or the jet, and there is therefore no component of force holding the sheet against the blade other than the force exerted by the jet.

Coating weight applied with any given formulation is controlled by varying the force of the impinging jet, and by varying the thickness of the rigid blade. There are no moving parts in the coating unit.

Mr. Gunning also discussed formulations which have been used for coatings applied by this method, which has a range of $2^1/_2$ to 20 lb. per ream per side, over a wide range of formulation consistencies.

"The Comparison of the Air Doctor and Blade Coater," by G. L. Booth and N. Laughrey, Black-Clawson Co., Dilts Div., Fulton, N. Y., was the next paper, presented by Mr. Booth, who gave descriptions of presently available air doctors and blade coaters, patents, diagrams and photographs of these units. No attempt was made to compare one quality-wise versus another. Next described were the operation of air doctors, grades and products handled and results obtained. A similar comparison was made on blade coaters. There was a comparison of the two units in fields in which they overlap to show the advantage of one over the other and to show how to choose between the two basic methods.

"New Coating Preparation Plant at Blandin Paper Co." was the next paper, presented by C. A. Richardson, Blandin Paper Co., Grand Rapids, Minn. Mr. Richardson described the new clay unloading, mixing, and storage facilities, and of the coating preparation plant at the Blandin Paper Co.

Coating clay is unloaded by a vacuum system from hopper cars directly into a 50-ton batch make-down tank. It is then stored at 70% solids in agitated tanks.

In the coating preparation, 70% clay slip and starch slurry sufficient for a batch are screened into a mixing tank from which the mixture is pumped through a Hasco heater which brings the slurry instantaneously to the cooking temperature. The batch is collected in an agitated tank, and the plasticizer added. After adequate mixing, the batch is then pumped through another screen into a storage tank. From here, the coating is pumped at frequent intervals to the coater supply

Local Arrangements Committee: F. A. Strovink, American Cyanamid Co.; F. R. Marchetti, Titanium Pigment Corp.; R. E. Kilty, A. E. Staley Mfg. Co.; W. L. Craig, R. T. Vanderbilt Co., and H. L. Miller, Hercules Powder Co.

tanks from which it circulates through lines equipped with coolers to the coater.

Mr. Richardson also discussed the effect of several operating variables on the properties of the coating color.

"The Optical Efficiency of Titanium Dioxide Pigments in Paper Coatings" was presented next by F. R. Marchetti, Titanium Pigment Corp., New York, N. Y. W. R. Willets, of the same company, was co-author.

In contrast to filler applications where only very limited control can be exercised over the distribution of pigment on fiber surface, Mr. Marchetti pointed out that the paper coating formulator can do much to insure the proper conditions for maximum pigment effectiveness. Following a discussion of the factors which govern the optical efficiency of titanium dioxide pigments in paper coatings, the authors presented a theoretical basis and related it to commercial paper coatings.

Several paper coatings containing various combinations of titanium dioxide and low refractive index pigments were analyzed by the authors in terms of optical efficiency, and the brightening and opacifying properties of the coatings were explained in these terms. Examples were given of some of the conditions which can harm pigment efficiency by interfering with proper pigment distribution in the coating.

"Modification of Coating Color Flow Properties with Amines," by A. W. Claxton, Minerals & Chemicals Corp. of America, Menlo Park, N. J., was the final paper of the Monday afternoon session. Mr. Claxton stated that the addition of small amounts of certain amines or amine salts to clay-water slurries or to starch coating colors has a profound effect upon their rheological properties. Hagan rheograms of clay-water slurries containing varying quantities of diethylenetriamine illustrates an extreme degree of dilatancy, dependent upon the amount of amine used. The author offered a theory to explain the action of amines; a joining together of individual clay particles edge to edge to result in an effective increase in diameter to thickness ratio.

The extreme dilantancy exhibited in high solids slurries when polyamines were added was not evident in the starch coating colors studied. Instead, the coating colors show increased thixotropy. Leveling Index was increased from 0.299 to 0.413 in one case by use of 0.20% ethylenediamine.

Paper coated with colors containing amines show a significantly higher gloss, easier finishing, and smoother printing surface than control coatings without amines. Evaluation of coating colors with the roll-inclined plane equipment indicates that coating color solids can be reduced with little or no adverse effect upon a real ratio (a measure of the fluid

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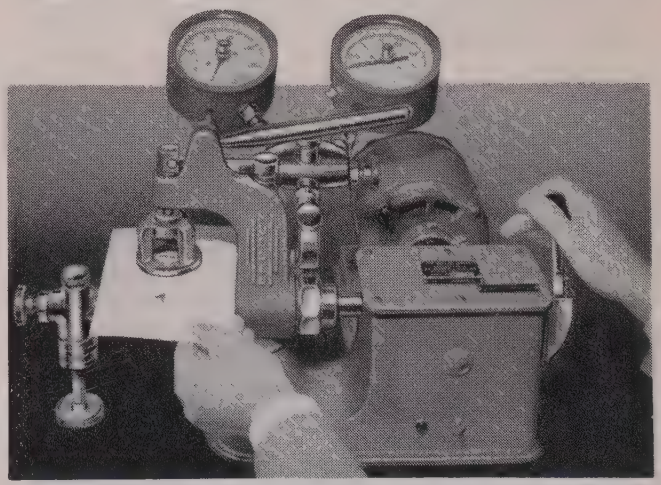

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First Session: P. W. Plante, St. Regis Co.; R. W. Hagemeyer, Wyandotte Chemicals Corp.; and R. C. Chase, University of Maine

migration properties of the coating color) and with accompanying improvement in gloss and printing smoothness.

Third Session

"Coatings, Past Present, and Future." On Tuesday morning, May 26, a panel symposium on the above subject was held under the chairmanship of A. W. Neubauer, Crown Zellerbach Corp., Camas, Wash., with presentations by the following panel:

N. I. Bearse, Oxford Paper Co., Lawrence Mass.—"Coatings —Past"
W. H. Aiken, Diamond-Gardner Corp., Middletown, O.—"Board Coating"
H. M. Annis, Oxford Paper Co., New York, N. Y.—"Coatings —Present"
P. J. Thoma, Time, Inc., Springdale, Conn.—"Coatings—Future"

Following the presentations by the panelists, a lively and stimulating question period was held. The text of Mr. Aiken's presentation appears in full elsewhere in this issue of *Tappi*.

Third Session: A. W. Neubauer, Crown Zellerbach Corp.; N. I. Bearse, Oxford Paper Co.; W. H. Aiken, Diamond-Gardner Corp.; H. M. Annis, Oxford Paper Co.; and P. J. Thoma, Time, Inc.

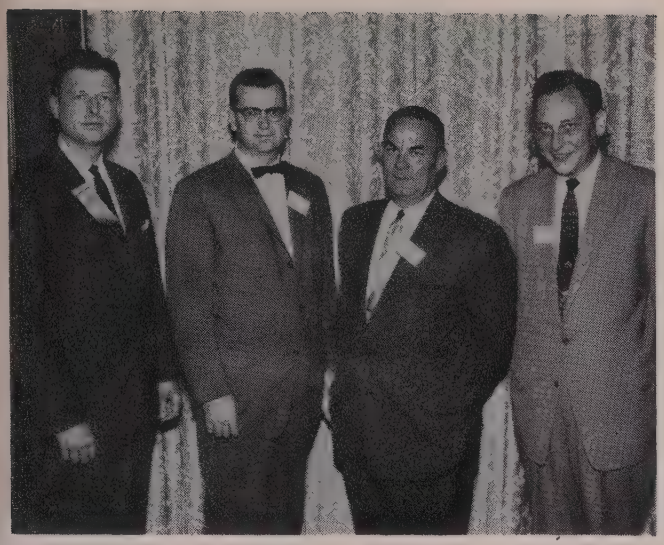
Fourth Session

After mill trips on Tuesday afternoon, the conferees assembled again on Wednesday morning, May 27, for the fourth technical session. Charles Whelpton, Champion Paper & Fibre Co., Hamilton, Ohio, was chairman of this session.

"The Effect of Styrene-Butadiene Ratio on the Properties of Clay Coated Paper," first paper of the fourth session, by R. F. Avery, A. R. Sinclair, and H. G. Guy, Koppers Co., Pittsburgh, Pa., was presented by Mr. Guy. A series of latices made with the same emulsifiers and polymerized under the same conditions varying only in styrene-butadiene ratios were tested as pigment binders for clay coated paper under controlled conditions. These latices were used alone and in combination with starch and casein. In all cases the total binder was 18% on clay solids. In combination with casein or starch the latex was reduced to one third of the binder. The styrene-butadiene ratio was varied from equal parts of styrene to butadiene to 90 parts of styrene and 10 parts butadiene in units of 10. These coatings were compared with those containing only starch or only casein, both at the 18% and 12% levels. Coatings were applied with the

Second Section: L. A. Gilbertson, American Can Co.; J. R. Gunning, Abitibi Power & Paper Co.; G. L. Booth, The Black-Clawson Co.; C. A. Richardson, Blandin Paper Co.; F. R. Marchetti, Titanium Pigment Corp.; A. W. Claxton, Minerals & Chemicals Corp. of America

Fourth Session: Charles Whelpton, Champion Paper & Fibre Co.; H. G. Guy, Koppers Co.; R. L. Hagerman, Dow Chemical Co.; C. R. Russell, U. S. Dept. of Agriculture; and D. S. Neif, National Starch Products, Inc.



Fifth Session: H. J. Connell, S. D. Warren Co.; W. L. Rhodes, Rochester Institute of Technology; A. Miller, Keceo Co.; and G. K. Cleek, Allied Chemical Corp.

Coating Council: C. G. Landis, Wica Chemicals, Inc.; F. Kaulakis, Consolidated Water Power & Paper Co.; J. T. Loomer, American Can Co.; J. E. Wilber, St. Regis Paper Co.; and R. W. Hagemeyer, Wyandotte Chemicals Corp.

Koppers-Dilts pilot plant coater. Wet rub resistance, pigment binding and gloss were all found to be affected by the styrene-butadiene ratio, with slight differences in stiffness, varnish holdout and fold resistance. No attempt was made to determine the cause of these differences. In all cases the addition of latex to the starch or casein system resulted in improved wet rub resistance. Increase of the styrene content tended to decrease the binding strength of the pigment binding, but the gloss increased.

"Study of the Water Retention of Latex Pigmented Coating Colors," second paper of the Wednesday morning session, by R. L. Hagerman, R. G. Jahn and W. H. Somers, Dow Chemical Co., Midland, Mich., was presented by Mr. Hagerman. This study of composition variables of latex bound colors on water retention included pigmentation, pigment dispersant composition and concentration, adhesive composition and level, solids content, and other additives. Water retention is related to the wet and dry pick resistance of boxboard coated with colors evaluated in the water retention work.

"Microparticle Size Vinyl Acetate Copolymer Emulsions as Economical, Low Odor, Clay Coating Binders," by N. G. Tompkins, Dewey & Almy Chemical Div., W. R. Grace & Co., Cambridge, Mass., was the third paper Wednesday morning. Mr. Tompkins described a number of experiments in which certain vinyl acetate copolymer emulsions were used as binders in clay or clay titanium dioxide coatings, both alone and in combination with starch. Results showed that certain of the emulsions tested offered an attractively economical means of obtaining coatings of high quality, with minimum odor and tendency to embrittle and discolor with age and exposure to light. The author listed suitable applications such as coating bread and frozen food wrapping material.

"High-Amylose Corn Starch—Properties and Prospects," by F. R. Senti and C. R. Russell, U. S. Department of Agriculture, Peoria, Ill., was the next paper, presented by Mr. Russell, who gave a progress report on high-amylose cornstarch to acquaint potential users with this new industrial type of starch which is still in the early developmental stages. The authors reviewed the cooperative program between plant breeders and chemists on the genetic increase of the amount of linear molecular amylose in cornstarch above the normal level of about 27%. To date analyses of breeding samples have disclosed starch containing up to 82% amylose; corn having starch of 50 to 60% amylose content could be available in quantity by 1960. Following a discussion of the properties of amylose and amylopectin—the linear and branched molecular components of starch. Mr. Russell described the properties of the high-amylose starches thus far studied and gave possible industrial uses for high-amylose starches and their chemical modifications for such fields as paper coating and food packaging.

Opening session of the Tenth Coating Conference

Coating Committee meeting

K. G. Chesley, The Crossett Co.; H. M. Annis, Oxford Paper Co.; J. T. Loomer, Continental Can Co.; S. W. Mead, Consolidated Water Power & Paper Co.

R. G. Macdonald, TAPPI; H. O. Ware, Beveridge Paper Co.; W. H. Aiken, Diamond-Gardner Corp.

"Cationic Starches as Paper Coating Binders," last paper of the fourth session, was written and presented by D. S. Grief, National Starch Products, Inc., Plainfield, N. J. Mr. Grief pointed out the need for high binding power and improved flow properties in binder systems to meet the present trend to high speed and lightweight coating. Since starch gives excellent flow characteristics it seems that study of starch modifications will bring further improvement in coating flow and facilitate binder reduction. In many instances some new modifications of starch, such as acetate and ether derivatives, have served to improve the above properties. The cationic starches are one of the most significant series of starch modifications to be introduced to the paper field in recent years. Some modifications have proved to be of great benefit to the paper industry as wet and additives due in part to their ability to bind securely to anionic surfaces such as cellulose fiber and to flocculate many substances such as pigments. The affinity of the cationic derivatives for cellulose could enhance coating anchorage and its tendency toward pigment flocculation. Cationic starches will be studied to determine the effect on water retention, strength, optical characteristics and high shear flow properties of the coating.

Fifth Session

The final session of the conference, on Wednesday afternoon, May 27, was chaired by H. J. Connell, S. D. Warren Co., Cumberland Mills, Me.

"Experience with Control Charts on Ink-Film Thickness," by W. L. Rhodes and J. Petrycki, Rochester Institute of Technology, Rochester, N. Y., was the first paper of this session. Mr. Rhodes presented this paper describing experience over a ten-month period with control charts on ink density and sharpness.

One of the experiments in color reproduction at Rochester Institute of Technology calls for repeating results obtained using an offset press to print three colors on a cast-coated stock. Density and sharpness are measured with a densitometer. The pressman is asked to achieve a constant ink density and sharpness within one sheet and from sheet-to-sheet on all press runs.

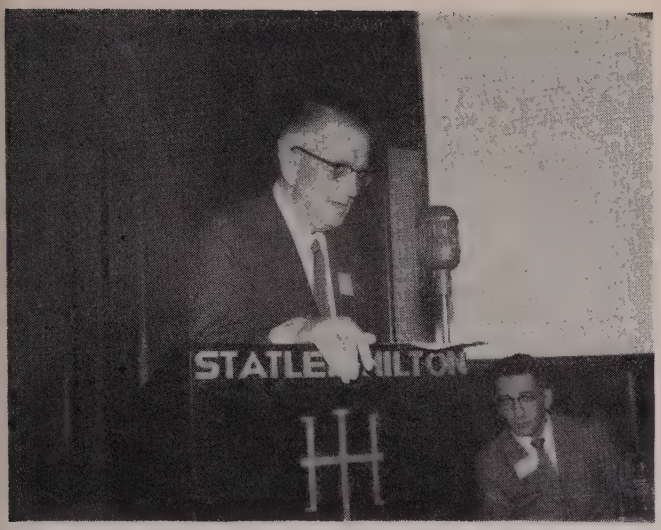
Mr. Rhodes described how the pressman tries to achieve uniformity and shows his results by means of control charts and related statistical tests.

One batch of paper exhibited a periodic sheet-to-sheet variation. Paper tests were run on these sheets to see if any of the tests methods correlated with press results.

"Penetration Controlling (or Water Retention) Properties of Colloid Additives," by A. Miller, Kelco., San Diego, Calif., was the second paper of the last session. Mr. Miller reported work on the holdout properties of aqueous solutions

F. Kaulakis, Consolidated Water Power & Paper Co.; R. G. Macdonald, TAPPI

R. W. Hagemeyer, Wyandotte Chemicals Co.; F. R. Marchetti, Titanium Pigment Corp.; H. O. Teeple, TAPPI



F. W. Frost, S. D. Warren Co., delivering the Edwin Sutermeister eulogy

S. W. Mead, Consolidated Water Power & Paper Co., luncheon guest speaker

when small amounts of colloid additives were added to the solution. As it had been observed that the addition of relatively small amounts of sodium alginate seemed to improve the holdout properties considerably a method was sought to measure this phenomenon accurately. "The Water Retention Test in Evaluating Coating Color" [*Tappi* 41: 77–79 (1958)] was adapted and it was found that very reproducible penetration times could be obtained. This test consists essentially of measuring under controlled conditions the time required for water to penetrate through a constant standard test paper from the aqueous coating. The end point is measured by a type of conductivity cell.

Solutions of various colloid additives were evaluated and it was found there was a considerable difference in the colloid's ability to hold out the aqueous system. Although viscosity of a solution is generally regarded as being the sole criterion of holdout of a solution, it was found that this is not necessarily true. Of more importance is the hold-out characteristic of the colloid itself. Some curves of penetration time vs. concentration for a number of colloids are shown to illustrate this point.

It was also found that in many cases the addition of an inorganic phosphate to a sodium alginate solution had a synergistic effect on increasing the hold-out time of the solution even though the viscosity decreased. This was found to be a function of degree of polymerization of the sodium alginate and the concentration of the added phosphate. None of the other colloid additives examined thus far has shown this type of synergistic effect.

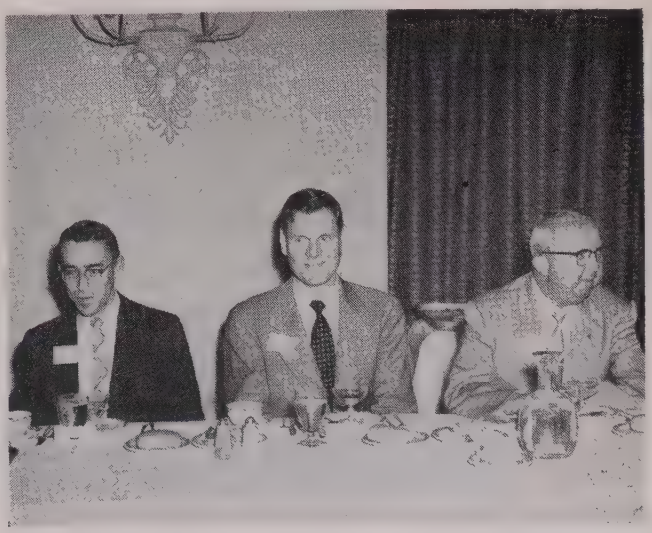
It was also found that between the pH values of 4 and 11.5 the penetration time of a sodium alginate solution is quite constant although there is a change of viscosity at the low pH level. This is interpreted to indicate that hold-out is a physical property of the colloid additive and probably due to the molecular and physical structure of the colloid.

"Rheological Properties of Suspensions and Polymeric Solutions," was the topic, presented by A. B. Metzner, University of Delaware, Newark, Del. Mr. Metzner reviewed the method of obtaining true flow curves from viscometric instruments, including the corrections necessary with both capillary tube and rotational instruments. The advantages of the greater flexibility of capillary rheometers over rotational instruments is seen to be counterbalanced by the greater ease and rapidity with which rotational data are obtained.

The final paper of the conference, presented by title, was "Application of Urea-Formaldehyde Concentrate 85 to Starch Clay Coatings on a High Solids Pilot Plant Roll Coater," by E. F. Birkett, Allied Chemical Corp., Hopewell, Va., and R. C. Chase, University of Maine, Orono, Me. An abstract of it follows:

The addition of this concentrate to the coating prior to application to the paper showed considerable improvement in the web resistance. Adequate wet rub resistance was de-

A. J. Winchester, TAPPI; G. E. Hall, Wyandotte Chemicals Corp.; and R. E. Kilty, A. E. Staley Mfg. Co.



J. E. Wilber, St. Regis Paper Co.; C. R. G. Maynard, Wiggins Teape Ltd.; K. G. Chesley, The Crossett Co.

veloped by all starches in the pH range of six to nine within the time available for drying at moderate speeds. As the coated paper cured, further wet resistance developed. The best results were obtained when using a waxy maize starch containing no amylose.

LUNCHEON

The official luncheon of the conference was held on Wednesday, May 27. J. T. Loomer, Coating Committee chairman, opened the proceedings and turned the meeting over to F. Kaulakis, general conference chairman, who introduced the head table guests. R. G. Macdonald, assistant to the president of TAPPI, spoke briefly on behalf of President J. R. Lientz, who was unavoidably absent. Greetings from the British Paper and Board Makers Association, Technical Section, were conveyed by C. R. G. Maynard, Wiggins Teape Group, England.

J. T. Loomer then introduced Stanton W. Mead, president, Consolidated Water Power & Paper Co., who delivered the official address of the banquet.

PLANT VISITS

On Tuesday afternoon, May 26, the technical sessions were recessed while the conferees visited plants in the Boston area. Three tours were available.

Tour A. Arthur D. Little, Inc. and Dewey and Almy Div., W. R. Grace & Co. 106 persons took this tour.

Arthur D. Little, Inc., Cambridge, Mass., was established in 1886 and is now the oldest and largest private research, engineering, and management consulting firm. The company's new Acorn Park laboratory facilities, begun in 1951, houses about two thirds of the total staff of just over 1000. Here scientists and engineers collaborate in a wide variety of research and development projects for clients, based on chemistry, physics, and biology and the related fields of engineering.

The company was originally founded as a paper research laboratory, and while now thoroughly diversified, it has maintained its interest in this area. Of particular interest to TAPPI visitors was the section featuring the development and improvement of paper-based products, and the coating laboratory containing a 20-in. wide coater which permits pilot scale production coating by knife, wire rod, roller, and dip methods at speeds from 400 to 500 f.p.m.

Dewey and Almy Chemical Div., W. R. Grace & Co., Cambridge, Mass., has a laboratory Roto Former and the new Merrill Brookfield High Shear Viscosimeter. Dewey and Almy have recently started to produce resins and part of this plant visit was an inspection of their new $500,000 polymer plant. Another interesting operation at Dewey and Almy that does not pertain to paper production is the production of meteorological balloons.

Tour B. Oxford Paper Co., Lawrence, Mass. 156 people visited the Oxford mill, which includes equipment and operations for the manufacturing of bleaching pulp and paper, double coated roll train high-speed coating and a new wide Eck supercalendar. Also available for inspection were certain mechanical paper handling and trimming operations.

Tour C. Nashua Corp., Nashua, N. H. This tour was taken by 105 persons.

The Nashua Corp. has some unusual operations such as heat seal coating and a special process for making heat-sensitive chart paper. Wet waxing and animal glue gummed tape production, as well as various letter and gravure printing operations were also open for inspection.

ELEVENTH COATING CONFERENCE

The Eleventh TAPPI Coating Conference will be held at the Edgewater Beach Hotel, Chicago, Ill., May 23–25, 1960.

Anyone wishing to offer a paper for the conference should contact R. W. Martinek, program chairman, Kimberly-Clark Corp., Neenah, Wis. General chairman of the conference is R. W. Hagemeyer, Wyandotte Chemicals Corp., Wyandotte, Mich. Suggestions regarding the program and arrangements for the conference will be welcomed by any member of the committee.

TAPPI Conferences

Tenth Testing Conference, Multnomah Hotel, Portland, Ore., Aug. 17–21, 1959.

Fiber Attraction and Bonding Symposium, The Institute of Paper Chemistry, Appleton, Wis., Sept. 9–11, 1959.

Ninth Corrugated Containers Conference, Jung Hotel, New Orleans, La., Sept. 15–17, 1959.

Fourteenth Paper-Plastics Conference, Edgewater Beach Hotel, Chicago, Ill., Sept. 21–23, 1959.

Fourth Dinking Conference, Roger Smith Hotel, Holyoke, Mass., Oct. 8–9, 1959.

Fourteenth Engineering Conference, Penn-Sheraton Hotel, Pittsburgh, Pa., Oct. 12–15, 1959.

Thirteenth Alkaline Pulping Conference, Robert Meyer Hotel, Jacksonville, Fla., Nov. 4–6, 1959.

PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

Loren F. Ashwood, Plant Manager, Downing Box Co., Milwaukee, Wis., a 1939 graduate of University of Illinois.

Edward Bearman, Laboratory Manager, Morningstar, Paisley, Inc., New York, N. Y., a 1952 graduate of New York University.

Julius Benko, Lignin Chemist, Research Group Leader, Sulphite Pulp Manufacturers' Research League, Appleton, Wis., a 1950 graduate of Technical University, Graz, Austria.

Herbert F. Berger, Regional Engineer, National Council for Steam Improvement, Baton Rouge, La., a 1950 graduate of Rutgers University.

William C. Black, Chemist, Penick and Ford, Ltd., Inc., Cedar Rapids, Iowa, a 1932 graduate of Iowa State College.

John W. Bodey, Mill Manager, Deynsham Paper Mills Ltd., Bristol, England, a 1951 graduate of the University of Maine.

Richard L. Boegner, Chief Chemist, Lily-Tulip Cup Corp., Springfield, Mo., a 1944 graduate of Rockhurst College.

Howard J. Buechler, Sales Manager, Black-Clawson (Canada) Ltd., Montreal, Que., Canada, a 1948 graduate of Purdue University.

Thomas D. Callinan, Manager, Paper and Ink Research, International Business Machines, Corp., Yorktown Heights, N. Y., a 1939 graduate of St. Peter's College.

John A. Cheesman, Yonkers, N. Y., a 1955 graduate of New York State College of Forestry.

Bengt W. Collin, Head, Research Department, A/B Statens Skogsindustrier Forskningslaboratoriet, Djursholm, Sweden, a 1943 graduate of Royal Technical University.

Leon A. Cookman, Manager, Communications Services, International Business Machines Corp., Yorktown Heights, N. Y.

Richard H. Cubberley, Research Director, Research Laboratory, The Flintkote Co., Whippany, N. J., a 1923 graduate of the University of Pennsylvania.

John A. Dalton, Director, Towgood and Beckwith Ltd., Peterborough, England.

Kenneth C. Damp, Research Chemist, A. E. Staley Mfg. Co., Decatur, Ill., a 1955 graduate of Western Illinois State College.

Edward S. Davis, Program Manager, E. I. du Pont de Nemours & Co., Wilmington, Del., a 1943 graduate of Lehigh University.

Charles H. Driver, Director of Forest Research, International Paper Co., Bainbridge, Ga., a 1954 graduate of Louisiana State University with a Ph.D. degree.

Solis Dudnick, Chief Engineer, National Manufacturing Co., Kansas City, Mo., a 1943 graduate of University of Pennsylvania.

Fred N. Eastwood, District Supervisor of Sales, National Starch Products, Inc., New York, N. Y., a 1934 graduate of University of Mississippi.

Sven W. Englund, Staff Engineer and Consultant, Rayonier Inc., New York, N. Y., a 1943 graduate of Polytechnic Institute of Brooklyn, with a D.Ch.E. degree in 1951.

Carlos V. Escalona, Director of Research, P. R. Economic Development Administration, Roosevelt, Puerto Rico.

James C. Everett, Sales Engineer, American Shear Knife Co., Homestead, Pa. Attended Georgia Institute of Technology.

Glen D. Farrar, Turbine Specialist, General Electric Co., Atlanta, Ga., a 1935 graduate of Kansas State College.

Robert C. Fuehrer, Student, University of Maine, Orono, Me., a 1959 graduate.

Charles G. Gedeon, Technical Manager, Graphica, Commercial & Industrial Co., Beirut, Lebanon, a 1953 graduate of French University.

Sixto-E. Gimeno P., Technical Director, Papelera Uranga S. A., Tolosa, Guipuzcoa, Spain.

James R. Goots, Product Engineer, Technical Service, B. F. Goodrich Chemical Co., Cleveland, Ohio, a 1955 graduate of West Virginia University.

Ragnar Gundersen, Chief Chemist, Vestfos Cellulosefabrik A/S, Vestfossen Norway, a 1955 graduate of Technical University of Norway.

Peter J. Hakim, Consulting Engineer, Container Laboratories, Inc., New York, N. Y., a 1945 graduate of College des Freres, Rochefort, Belguim.

Niilo Hakkarainen, Assistant Superintendent, Sulphate Pulp Mill, Kemi Oy, Karihaara, Finland, a 1953 graduate of the Technical University of Helsinki.

Frank V. Halvonik, Group Leader, P. H. Glatfelter Co., Spring Grove, Pa., a 1950 graduate of University of Pittsburgh.

Henry Herman, Jr., Assistant Technical Superintendent, Container Corp. of America, Philadelphia, Pa., a 1949 graduate of Pennsylvania State University.

Kurt Heuer, Research Manager, Vereinigte Glanzstoff-Fabriken AG., Wuppertal-Elberfeld, Germany, a 1950 graduate of Gottingen University with a D.Sc. degree.

Robert A. Jackson, District Sales Manager, Chicago Bridge & Iron Co., Seattle, Wash., a 1937 graduate of University of Maryland.

John B. Kahoun, Project Engineer, Nekoosa-Edwards Paper Co., Port Edwards, Wis., a 1952 graduate of Milwaukee School of Engineering.

E. J. Kiddie, Production Manager, Owens-Illinois Glass Co., Paper Products Div., Jacksonville, Fla., a 1942 graduate of Oshkosh State Teachers College.

Karl Kocher, General Manager, Hermann Finckh Metalltuch- und Maschinenfabrik, Reutlingen/Wurtt, Germany, a 1910 graduate of Technical Institute.

Frederick E. Labrom, Mill Manager, Papyrus Pty. Ltd., Sydney, Australia.

Rodolfo Lamine, Mill Manager, Cartiere Ambrogio Binda, Milano, Italy, a 1953 graduate of Polytechnic Institute of Milano.

William F. Linke, Group Leader, American Cyanamid Co., Stamford, Conn., a 1945 graduate of City College of New York, with a Ph.D. degree from New York University in 1948.

Jack W. Long, Research Chemist, Potlatch Forests Inc., Lewiston, Idaho, a 1956 graduate of Montana State College.

Helen Malmstrom, Librarian, Skogsbiblioteket (The Forestry Library), Stockholm, Sweden.

David F. Mason, Director of Research & Development, Southern Dyestuff Co., Div. of American-Marietta Co., Charlotte, N. C., a 1936 graduate of Emory and Henry College.

Wladimiro Menzinger, Managing Director, Talleres Coghlan S. A., Buenos Aires, Argentina.

George Morley, Project Engineer, Black-Clawson Co., Inc., Watertown, N. Y.

Herbert C. Nelson, Hydraulic Engineer, Kimberly-Clark Corp., Neenah, Wis., a 1953 graduate of University of Minnesota.

John M. Nevin, Lieutenant, U. S. Air Force, Edwards Air Force Base, California, a 1957 graduate of Princeton University.

Sven-Ake Nyhren, Engineer, AB Montoil, Stockholm, Sweden, a 1953 graduate of Technical College, Stockholm.

John J. O'Keefe, Jr., Salesman, Dow Chemical Co., Boston, Mass., a 1950 graduate of Boston College.

Alfred G. Olson, Sales Engineer, Beloit Iron Works, Beloit, Wis., a 1936 graduate of Armour Institute.

Robert J. Olson, Research Chemist, General Foods, Ltd., Cobourg, Ont., Canada, a 1955 graduate of University of Saskatchewan.

Mark F. C. Paige, Librarian, Imperial Chemical Industries Ltd., Stevenson, Ayrshire, Scotland.

David H. Payor, Chief Consumer's Representative, Scott Paper Co., Sandusky, Ohio, a 1954 graduate of Bowdoin College.

J. W. Perry, Librarian, The University of Natal, Durban, South Africa.

Clyde A. Plaskett, Group Leader, St. Regis Paper Co., Yonkers, N. Y., a 1950 graduate of University of Wisconsin.

Barrie T. Reed, Industrial Engineer, Union Bag-Camp Paper Corp., New York, N. Y., a 1958 graduate of Columbia College.

Raymond N. Riding, Technical Service Representative, West Virginia Pulp and Paper Co., New York, N. Y.

Leto Ridi, Technical Director, Istituto Poligrafico dello Stato, Rome, Italy, a 1932 graduate of University of Naples.

Hugh Y. Rienhoff, Secretary-Treasurer, Alloy Cladding Co., Inc., Baltimore, Md., a 1954 graduate of Johns Hopkins University.

S. M. Robertson, District Sales Manager, Koppers Co., Inc., Plastic Div., Los Angeles, Calif., a 1948 graduate of Cornell University.

Heizaburo Saito, Staff Engineer, Diashowa Pulp & Paper Co., Shizuoka, Japan.

John R. C. Sheldon, Assistant General Manager, Aylesford Paper Mills, Albert E. Reed Co., Kent, England, a 1949 graduate of Cambridge College.

Brent L. Smith, Manager, White Electric Motors, Lowell, Mass.

James L. Snaman, Vice-President, The Pannier Corp., Pittsburgh, Pa., attended the University of Pittsburgh.

William A. Snyder, Project Engineer, Scott Paper Co., Chester, Pa., a 1956 graduate of Lehigh University.

Leo J. Stage, Technical Director, Arvey Corp., Jersey City, N. J., a 1934 graduate of New York University, College of Engineering.

Ernest G. Steinhauer, Research Engineer, West Virginia Pulp and Paper Co., Covington, Va., a 1946 graduate of Institute of Technology, Zurich, Switzerland.

John D. Wallace, Manager, Heavy Chemicals Sales Div., Dow Chemical of Canada, Ltd., Sarnia, Ont., Canada, a 1944 graduate of University of Toronto.

Peter G. Zouck, President, Alloy Cladding Co., Inc., Baltimore, Md., a 1941 graduate of Harvard University.

TAPPI Notes

Roland L. Allen, Jr., formerly with J. R. Sirrine Co., is now Vice-President of Eastern Engineering Co., Atlanta, Ga.

William E. Anderson is now Assistant Superintendent of the Converting Dept., Riegel Paper Corp., Milford, N. J.

George H. Andrews, Jr., The Mead Corp., has been transferred from Chillicothe, Ohio, to Sylva, N. C., as Manufacturing Control Supervisor.

Fred W. Argue is now Executive Vice-President, Stone & Webster Engineering Corp., Boston, Mass.

Nicholas Bachynski, formerly with Reliance Electric & Engineering Co., is now an Engineer with Chas. T. Main, Inc., Boston, Mass.

John H. Barrette, formerly Mill Chemist, is now Assistant Superintendent, Yard and Fiber Production, Biron Div., Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

David R. Bernhardt, formerly with Oxford Miami Paper Co., is now Process Control Supervisor, Eastern Fine Paper & Pulp Div., Standard Packaging Corp., Bangor, Me.

Carl Botkin, formerly Chief Chemist, Charles Bruning Co., Inc., Teterboro, N. J., is now Vice-President in charge of Research.

Francis O. Boylon, formerly Resident Manager, Crown Zellerbach Corp., Camas, Wash., has been transferred to San Francisco, Calif., as Assistant Vice-President.

C. Edwin Brandon, formerly Associate Professor, is now Director of Pulp and Paper Technology, Miami University, Oxford, Ohio.

Fred L. Buchanan, formerly with the National Gypsum Co., is now Technical Superintendent, Central Fibre Products Co., Quincy, Ill.

John S. Buchanan, formerly Research Chemical Engineer, Nekoosa-Edwards Paper Co., Port Edwards, Wis., is now Technical Assistant to the Kraft Mill Superintendent.

Lewis K. Burnett, formerly Manager, Product Coordination and Procurement, is now Assistant to the Senior Vice-President, Ohio Boxboard Co., Rittman, Ohio.

Hugh D. Burnie, formerly Mill Chemist, is now Superintendent, Coating Dept., Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Robley A. Butler is now Resident Manager, Crown Zellerbach Corp., Camas, Wash.

Josiah Chapman is now Head, Process Dept., Central Laboratory, Albert E. Reed & Co. Ltd., Larkfield, Nr. Maidstone, Kent, England.

C. Andre Charron, formerly a student at the University of Maine, is now with Stevens Paper Mills, Westfield, Mass.

Jean Y. Chartrand, Chief Chemist, Rolland Paper Co. Ltd., St. Jerome, Que., has been transferred to Mont-Rolland, Que.

James K. J. Cheng, formerly with Nekoosa Edwards Paper Co., is now Senior Research Chemist, National Cash Register Co., Dayton, Ohio.

Ernest C. Cowell, formerly with West Virginia Pulp & Paper Co., is now Staff Assistant to the Director of Quality Control and Sales Service, Allied Paper Corp., Kalamazoo, Mich.

Richard C. Crain is now Technical Director for Rhinelander Paper Co., Rhinelander, Wis.

Alfred H. Croup is now Development Physicist, Research and Development Section, Hammermill Paper Co., Erie, Pa.

Eugene P. de Prosse, formerly with Tecnica Cubana S. A., is now Process Engineer, Lafayette Engineering Div., Fibreboard Paper Products Corp., Lafayette, Calif.

Theodore W. Dunn, formerly President, Dunn Paper Co., Port Huron, Mich., is now Chairman of the Board and Treasurer.

Robert A. Dykes, formerly with P. H. Glatfelter Co., is now an Officer Candidate, U. S. Navy, Newport, R. I.

George A. Farrah, of the National Container Corp., has been transferred from Tomahawk, Wis., to Chicago, Ill., as Plant Manager.

James F. Fenske is now Manager, Industrial Sales Dept., Allis-Chalmers Mfg. Co., Norwood, Ohio.

Alexander F. D. Ferguson, Special Student at The Institute of Paper Chemistry, is returning to his position as Department Manager, R. & W. Watson Ltd., Renfrewshire, Scotland.

David W. Flotow, formerly with Hercules Powder Co., is now a Research and Development Project Engineer with Consolidated Paper Co., Monroe, Mich.

George E. Fromm, American Cyanamid Co., has been transferred from Chicago to Mobile, Ala., as Southern Regional Manager, Paper Chemicals Dept.

Robert C. Fuehrer, formerly student, University of Maine, is now with the Spaulding Fibre Co., Milton, N. H.

John D. Gaffney is now Manager, Technical Service and

Product Development Dept., Rhinelander Paper Co., Rhinelander, Wis.

William W. Graham, formerly Laboratory Group Leader, is now a salesman for E. I. du Pont de Nemours & Co., Inc., Chicago, Ill.

Joseph J. Hackett, formerly with office Electronics, Inc., is now President of J. J. Hackett & Co., Chicago, Ill.

Donald D. Hall, formerly with the E. B. Eddy Co., is now Manager, Production Services, Eastern Fine Paper & Pulp Div., Standard Packaging Corp., Bangor, Me.

Raymond S. Hatch, retired from Hudson Pulp & Paper Corp., is now Consultant in Pulping, Bleaching and Papermaking, Menlo Park, Calif.

Henry R. Hutt, Salesman, Dow Chemical Co., has been transferred from Atlanta, Ga., to Charlotte, N. C.

Joseph E. Hartford, Vice-President and General Manager, Paper Machine Div., The Black-Clawson Co., Inc., has been transferred from Middletown, Ohio, to Watertown, N. Y.

H. Maxwell Hughson is now Technical Manager, Biron Div., Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Paul A. Jones, formerly student, University of Maine, is now Control Engineer, Oxford Paper Co., Rumford, Me.

Joseph W. Judge, Jr., Riegel Paper Corp., has been transferred from Milford, N. J., to Hughesville, N. J., as Assistant Mill Superintendent.

Harold E. Keene, formerly with Goslin-Birmingham Mfg. Co., Inc., is now Sales Engineer with Improved Machinery Co., Birmingham, Ala.

Anders Kellgren, American Defibrator Co., has been transferred from New York, N. Y., to AB Defibrator, Stockholm, Sweden.

Krishna K. Khandelwal, formerly with Dewey & Almy Chemical Div., W. R. Grace & Co., is now Senior Development Engineer with Darak Battery Separator Laboratory, Cambridge, Mass.

William A. Kirkpatrick, consultant, is now President of the Kalamazoo Paper Box Co., recently merged with the Acme Folding Box Co., Kalamazoo, Mich.

Erwin L. Kischel, formerly Technical Director, Alpha Cellulose Corp., Bloomington, Ill., is now Comptroller.

Jurgen Kleinau, formerly Technical Trainee, Bergstrom Paper Co., Neenah, Wis., is now Technical Assistant.

Aage M. Leth, Consultant, is now Supervisor of Technical Service, Celulosa de Chihuahua S. A., Mexico, D. F.

Joseph L. McCarthy, formerly Professor of Chemical Engineering, University of Washington, Seattle, Wash., is now Dean of the Graduate School.

Amos J. Miner is now Midwest Regional Sales Manager, Dewey & Almy Chemical Div., W. R. Grace & Co., Chicago, Ill.

William A. Moggio is now Production Manager, Lake States Yeast Div., Rhinelander Paper Co., Rhinelander, Wis.

Theodore W. Montag, Jr., formerly with the Brown Co., is now Process Engineer, Sulphite Pulp Mill, Alaska Lumber & Pulp Co., Inc., Sitka, Alaska.

Thomas C. Morganstern, formerly with A. E. Staley Mfg. Co., is now Regional Technical Sales Representative for Minerals & Chemicals Corp. of America, Menlo Park, N. J.

William A. Morton, Assistant Research Manager, Dewey & Almy Chemical Div., W. R. Grace & Co., has been transferred from Chicago, Ill., to Cambridge, Mass.

William R. Moulton, West Virginia Pulp & Paper Co., has been transferred from Keyser, W. Va., to Luke, Md., as an operating engineer.

Merle L. Noyes is now Technical Assistant to the Superintendent of the Converting Dept., Riegel Paper Corp., Milford, N. J.

George A. Oechsle, Jr. is now Plant Manager, Continental Can Co., Robert Gair Div., Elkhart, Ind.

T. Proctor Pace, formerly engineer with Continental Can Co., Inc., Greenville, S. C., has been transferred to Augusta, Ga.

Robert J. Pfeiffer, formerly with Charles Bruning Co., Inc., is now Quality Control Manager with Standard Packaging Corp., Clifton, N. J.

Malcolm B. Pineo, formerly with Brunswick Pulp & Paper Co., is now Production Manager, Hollingsworth & Whitney Div., Scott Paper Co., Mobile, Ala.

Gordon W. Pixler, formerly with Diamond Alkali Co., is now Supervisor, Paper Technical Service, Colton Chemical Co. Div., Air Reduction Co., Cleveland, Ohio.

Francis Ratliff is now Research Director, Rhinelander Paper Co., Rhinelander, Wis.

Giovanni Rattalino, Research Chemist, Cartiere Burgo, has been transferred from Turin, Italy, to Romagnano Sesia (Novara), Italy.

Mervin C. Rogers, formerly with R. R. Donnelley & Sons Co., is now Graphic Arts Consultant and Director, Chicago Paper Testing Laboratory, Chicago, Ill.

Robert S. Savage, Goodyear Tire & Rubber Co., has been transferred from Needham Heights, Mass., to Akron, Ohio, as Senior Sales Engineer, Chemical Div.

James Schad is now Technical Supervisor, Biron Div., Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Henry Schreier, Chief Steam and Power Engineer, Hudson Pulp & Paper Corp., has been transferred from Palatka, Fla., to New York, N. Y.

Ronald R. Schutts, formerly with Robert Gair Div., Continental Can Co., is now Chemist, Central Technical Dept., St. Regis Paper Co., Deferiet, N. Y.

David M. Shapleigh is now Eastern Division Manager, Bauer Bros. Co., Springfield, Ohio.

Francis J. Sloan, formerly with Package Machinery Co., is now Consultant at Springfield, Mass.

C. Wayne Smith is now Vice-President of Inland Container Corp., Rome, Ga.

Alexander C. Sutherland, Technical Sales Representative, E. I. du Pont de Nemours & Co., Inc., has been transferred from Wilmington, Del., to Philadelphia, Pa.

John H. Symes, formerly with National Container Corp. of Virginia, is now Chemist, Polk County Air Pollution District, Florida State Board of Health, Winter Haven, Fla.

James S. Tipka, formerly with Scott Testers, Inc., is Technical Sales Representative, Boler Petroleum Co., Ardmore, Pa.

Charles R. Tittemore is now Assistant General Manager, Anglo-Newfoundland Development Co. Ltd., Grand Falls, Nfld.

Roger L. Traxler, formerly with the Marinette Paper Div., Scott Paper Co., has been transferred from Fort Edward, Wis., to Chester, Pa., as Process Control Engineer.

Richard T. Trelfa, Perkins-Goodwin Co., has been transferred from Kalamazoo, Mich., to New York, N. Y., as Vice-President and Director.

Charles S. Waddell, formerly with Abitibi Power & Paper Co. Ltd., is now Chief, Field Service Engineering, Kenwood Mills Ltd., Arnprior, Ont., Canada.

William E. Welliver, formerly with New York & Pennsylvania Co., Inc., is now Technical Sales Representative, Minerals & Chemicals Corp. of America, Menlo Park, N. J.

William J. White is now Manager, Technical Sales Service, Mosinee Paper Mills Co., Mosinee, Wis.

Dan B. Wicker is now Vice-President and Director of Research and Development, Huyck Felt Co., Rensselaer, N. Y.

Robert C. Williams, formerly with Gardner Board & Carton Co., is now Group leader of Research, Albemarle Paper Mfg. Co., Richmond, Va.

Kurt A. Wohl, formerly with General Motors Corp., is now Project Engineer, Thatcher Glass Mfg. Co., Elmira, N. Y.

Piotr Zenczak, Evans Product Co., has been transferred from Coos Bay, Ore., to Corvallis, Ore., as Plant Manager.

Thomas G. Zentner, Gardner Board & Carton Co., has been

transferred from Lockland, Ohio, to Middletown, Ohio, as Director of Research and Development.

* * *

H. Reeve Angel & Co., Inc., has moved from New York, N. Y., to 9 Bridewell Place, Clifton, N. J. *Thomas L. Harrocks, Jr.*, is their corporate representative in TAPPI.

The name of the Mead-Atlanta Paper Co., Atlanta, Ga., has been changed to Mead Packaging, Inc., Atlanta Paper Co. Div. *William Conner* is their corporate representative.

Dr. Neubauer has succeeded *R. S. Jobin* as the corporate representative in TAPPI of the Tasman Pulp & Paper Co. Ltd., Auckland, N. Z.

* * *

John I. Geary has succeeded *John F. Campbell* as the official representative in TAPPI for Barrett Div., Allied Chemical Corp., Sunbury, Pa.

Bensing Bros. & Deeney, Philadelphia, Pa., has assumed the name of its parent company, Sun Chemical Corp., and has moved to 750 Third Ave., New York 17, N. Y. *Sidney Shapiro* continues as their official representative in TAPPI.

Ciba Co., Inc., has moved from New York, N. Y., to Route 208, Fair Lawn, N. J. *Charles Weidmann* is their official representative in TAPPI.

H. A. Lips succeeds *K. C. Johnson* as the official representative in TAPPI for E. I. du Pont de Nemours & Co., Inc., Dyes and Chemicals Div., Wilmington, Del.

National Aluminate Corp., Chicago, Ill., has changed the company name to NALCO Chemical Co. *R. E. Eddy* continues as their official representative in TAPPI.

William W. Moore, Vice-President in charge of Sales, succeeds *H. M. Pier* as the official representative in TAPPI for Research-Cottrell, Inc. The mail address has been changed from New York, N. Y., to Bound Brook, N. J.

The J. O. Ross Engineering Div., Midland-Ross Corp., has moved from 444 Madison Ave. to 730 Third Ave., New York 17, N. Y. *S. W. Fletcher* is their official representative in TAPPI.

The Texas Co., Port Arthur, Tex., has changed its name to Texaco, Inc., effective May 1, 1959.

Industry Notes

Production

Production of paper and board for the first four months of 1959 is estimated at 11,075,000 tons, 11.5% above the year-ago total.

Production of newsprint during the first quarter of 1959 totaled 460,000 tons, about 21,000 tons over the 1958 first quarter level, and 1% below the 1957 first quarter high of 464,500 tons. Fine paper production for the first quarter of 1959 showed a 9% increase over the same period of 1958. Within the fine paper group, chemical wood pulp writing paper rose 9.7% and bristols rose 10.6%. An even greater percentage increase over 1958 levels was shown in production of book paper, which rose 12.4%. Production of coarse paper for the first quarter of 1959 exceeded 1958 first quarter output by 8.3%, or 74,000 tons. Within this category, shipping sack, bag, and other converting paper showed the greatest improvement over 1958 levels. Total sanitary tissue production for the first quarter of 1959 was 8.7% above 1958 production for the like quarter, while special industrial paper production was 176,000 tons, representing a 32,000-ton increase over the first quarter of 1958.

An examination of the first quarter 1959 data on paperboard production reveals that container board, which accounts for over half of total paperboard output, was 12% above production for the first quarter of 1958 and 0.2% above output for the same period of 1956—the year when container board output reached its historical high. First quarter output of bending board, which includes special food board, advanced to a level 9% above that of the corresponding quarter of 1958. Production of nonbending board totaled 282,000 tons, representing an 18% increase over first quarter 1958 output.

Although total paper and paperboard production during the first quarter of 1959 was high relative to the first quarter of the preceding year, a few paper and paperboard grades did not follow this trend. Wet machine board fell 6.5% from first quarter 1958 output, paper-machine coated printing and converting paper declined 1.4% below 1958 production, and glassine, greaseproof and vegetable parchment declined by 1.6%. But the great majority of the individual grades showed major gains from year-ago levels, and total output of paper and paperboard in the first quarter of 1959 established a new three-month high.

Pulpwood

Total receipts of pulpwood for the first quarter of 1959 were 9,177,000 cords, an increase of 401,000 cords over receipts for the comparable quarter of 1958. Domestic receipts were 490,000 cords above the year earlier total, while imports declined 89,000 cords. The South Atlantic region was the only region showing a decrease in receipts of wood pulp, declining 23.9% below the first quarter of 1958.

Pulpwood consumption in the first quarter of 1959 was 9,361,000 cords, an increase of 631,000 cords over the same period of 1958. The Northeastern region showed a 1% decline in consumption while all other regions showed increases ranging from 3% in Western regions to 11% in North Central and South Atlantic region.

Inventories of pulpwood at the end of March were 5,736,000 cords, a decrease of 116,000 cords below February, 1959. This represents a 14% decline from the inventory level at the close of the first quarter of 1958. The fall off from February, 1959, levels ranged from 1% in the North Central region to 8% in the South Atlantic region. Inventories in the South Central region increased slightly over the February, 1959, level of 517,000 cords.

Wastepaper

March wastepaper receipts were 801,000 tons, for the first quarter of this year they totaled 2,477,201 tons, an increase of 455,201 tons above receipts for the first quarter of last year.

Wastepaper consumption for March was 794,431 tons; the January–March total was 2,253,000 tons, an increase of 197,430 tons above consumption for the first quarter in 1958.

Inventories at the end of March were 455,796 tons, an increase of 6,000 tons above February and a decrease of 40,000 tons below inventories at the end of February, 1958.

Consumption of total fibrous materials for March was 887,000 tons; the January–March total was 2,513,000 tons, an increase of 205,663 tons above consumption for the first quarter of 1958.

Inventories of total fibrous materials at the end of March were 917,000 tons, a decrease of 19,000 tons below February, and a decrease of 93,000 tons below inventories at the end of March, 1958.

Wood Pulp

Production of wood pulp for the first quarter of 1959 was 5,837,000 tons, an increase of 491,000 tons above the comparable quarter of 1957.

Wood pulp consumption in the manufacture of paper and board for the first quarter of 1959 was 6,034,000 tons, an increase of 579,000 tons above consumption for the first quarter of 1958.

Imports of wood pulp for January–March, 1959, were 582,402 tons, an increase of 23% above imports for the

first quarter of last year. Imports from Canada for the first quarter of 1959 were 472,251 tons, an increase of 18% over the comparable quarter of 1958. Overseas imports for the first quarter of 1959 were 109,131 tons as compared to 76,219 tons in the first quarter of 1958.

Exports of wood pulp for the first quarter of this year were 143,914 tons, as against 139,772 tons exported during the same period last year.

Inventories of wood pulp at paper and board mills at the end of March were 547,000 tons a decrease of 1000 tons below inventories at the end of March and 11,000 tons below inventories at the end of February, 1958.

Fiber Boxes

Annual demand for corrugated and solid fiber shipping containers is forecast at a level of 2 billion sq. ft. a week for 1959, an 8% increase over shipments in 1958, Peter W. Hoguet, president, The Econometric Institute, New York, told members of the Fibre Box Association assembled at their 20th Spring Meeting in Washington, D. C.

Mr. Hoguet further predicted that the demand for fiber boxes will rise an additional 3% in the first quarter of 1960. He stated that he felt that industrial production will continue to rise throughout 1959, though at a slower rate than in the second half of 1958. Mr. Hoguet pointed out that one of the most outstanding trends in increased fiber box demand over the years is the use of these versatile shipping containers to package consumer durable goods. In the 1951–52 period only 6% of the total output of the fiber box industry was used for consumer durable goods. In the 1954–57 period however, 14% of the total production of fiber boxes went into consumer durable goods industries. He also said that the construction materials industry which used only 1.8% of fiber box output in 1951–52 has increased to 4.3% in the 1954–57 period.

Corrugated

Shipments of corrugated boxes during the first quarter of 1959 averaged 1.9 billion sq. ft. per week—14.5% higher than the same period of 1958 and 8% better than 1957, P. L. Brockington, assistant statistician, Fibre Box Association told the Association members assembled at the 20th Spring meeting in Washington, D. C.

Mr. Brockington distributed the 1958 Fibre Box Industry's Statistical Yearbook which showed shipments in 1958 reaching 97.3 billion sq. ft. This is the second best year the industry has ever had being topped only by 1956 shipments of 97.6 billion sq. ft. Mr. Brockington further stated that if the industry continues to produce at the same rate that they could expect a record year of more than 105 billion sq. ft. in 1959.

Wood Industries Conference

The A.S.M.E., F.P.R.S., A.I.C.E. Wood Industries conference is scheduled for Portland, Ore., Sept. 10–12, 1959. This conference is being sponsored jointly by the American Society of Mechanical Engineers, Wood Industries Division; Forest Products Research Society, Pacific Northwest and Inland Empire Sections; and the American Institute of Chemical Engineers, Washington-Oregon Section.

Through the combined efforts of the above three organizations, a broad gage, first-class program is being assembled for this national (plus Canada) forest products meeting. Attending this conference will be operating, technical, and management people representing a wide diversity of interests in all phases of forest products.

Employment

Employment in the New York-Northeastern New Jersey metropolitan area rose 19,000 in April, as increases in construction service, and other nonfactory industries more than offset declines in manufacturing, Herbert Bienstock, Acting Regional Director of the U. S. Department of Labor's Bureau of Labor Statistics announced today. The April increase raised the area's job total to 5,432,000, about 25,000 above the year-ago level.

Saare Medal

Thomas J. Schoch, Ph.D., supervisor of basic starch chemistry at the Corn Products Co.'s George M. Moffett Research Laboratories, Argo, Ill., has been awarded the Saare Medal for his pioneering research work in the field of starch and starch fractions.

Mr. Schoch received the medal during the 1959 Starch Congress held recently in Detmold, Germany. The first

Presentation of Saare Medal to T. J. Schoch (right) by L. Altrogge at Detmold, Germany, April 21, 1959

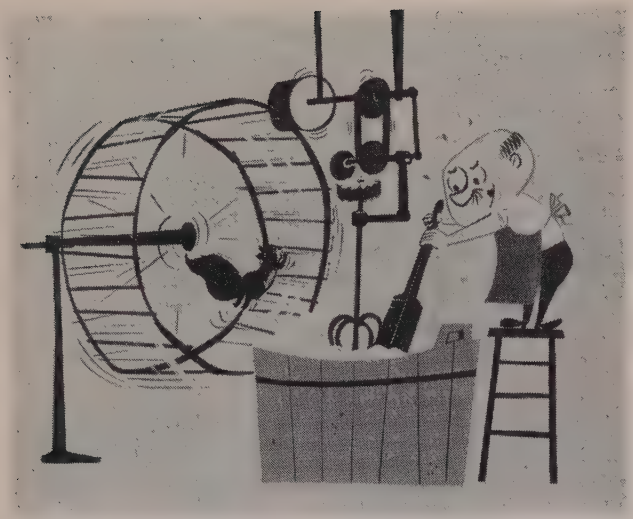
paper delivered at this congress was Mr. Schoch's, "Measuring the Useful Properties of Starch."

Presentation of the Saare Medal was made by L. Altrogge, president of the Association of Cereal Research, before several hundred carbohydrate chemists and starch technologists from all parts of Europe. The Saare Medal, named in honor of Professor Doctor Oskar Saare, founder of modern starch research in Germany, is awarded each year to one man who has made outstanding achievements in the field of starch research. The selection is made by the executive committee of the Association of Cereal Research.

Consolidated Water Power

William O'Gara has been appointed assistant production manager of the Wisconsin Rapids Division of Consolidated Water Power & Paper Co., it had been announced by L. W. Murtfeldt, division manager.

Mr. O'Gara, who has been serving as supercalender and winder superintendent since 1945, joined the company in 1942. He is a native of Spooner, Wis., and holds a B.A. degree from the University of Wisconsin.



"Early paper production," intoned the narrator as the above cartoon flashed on the screen. The drawing was art of a 50-slide presentation on the history of papermaking given by Simpson Paper Co., Everett, Wash., as its portion of the recent "Paper On Parade" symposium by Blake, Moffitt & Towne, San Francisco. Straight and factual narration by Simpson's Dick Denman, administrative assistant for sales, was matched to the spoofing cartoons. The incongruous approach gave the presentation a refreshing appeal. Graphic arts audiences ranging up to 600 persons attended "Paper on Parade" sessions in Seattle, Portland, San Francisco, and Los Angeles

George Jackson has been named by Mr. Murtfeldt to succeed Mr. O'Gara. Mr. Jackson began his employment with the company in 1935 and has been a super and winder tour foreman since 1946.

WYOMISSING

Wyomissing Paper Products (division of The Narrow Fabric Co., Reading, Pa.) has awarded a contract for design, engineering, and construction of a new 150,000 sq. ft. plant to William F. Lotz, Inc., Philadelphia construction firm.

Wyomissing is a leading producer of flint and glazed papers (Wyomissing, Reading, and United brands) for use in set-up and folding boxes, phonograph record labels, and other packaging requirements and also of cloth-lined papers for maps and heavy-duty purposes. The Narrow Fabric Co., with which Wyomissing merged in June 1957, is one of the nation's leading producers of narrow textiles including elastic and nonelastic, braided, woven, knitted, and lace products.

According to Samuel R. Fry, president, the new facility is designed to modernize and better integrate all production and materials handling functions on an efficient straight line basis. The transition to the new plant will be made without interrupting production by utilizing the facilities of the two existing plants throughout the entire changeover.

The new structure, to be erected on an 80-acre tract a few miles north of Reading, will incorporate a cantilever design. The cantilever structural design will enable a concentrated weight load to be carried at any point. This design also made possible a 15 to 20% saving in steel. Cantilevering further affords flexibility in adding monorail or hoist systems at any point in the building at any future time.

According to Mr. Fry, the primary purpose of the move is to modernize the company's facilities and to allow for future expansion on an efficient straight-line basis. The site was chosen because of its easy access to all Eastern metropolitan markets; it will have its own railroad siding and is also convenient for truck shipment via high-speed turnpikes.

New manufacturing building for Wyomissing Paper Products Div., Narrow Fabric Co., Reading, Pa.

J. C. WILSON

J. C. Wilson Ltd. at Lachute, P. Q., have recently rebuilt one of their fourdrinier machines to increase their production and allow a wider variety of grades.

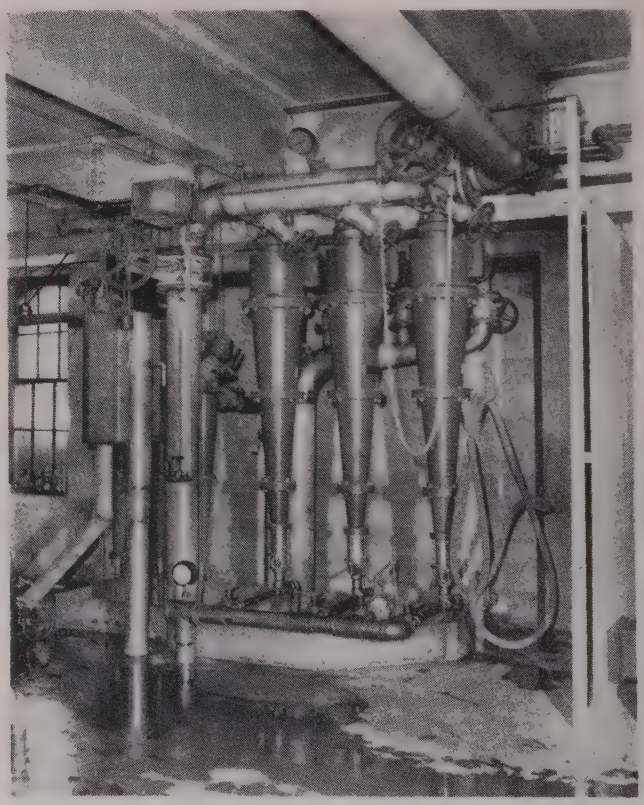
This mill is a kraft specialty mill making a variety of specialty papers, both bleached and unbleached. They have their own converting plant where a large part of their production is converted into bags and towels.

The mill first started in 1880 and at that time, four machines were installed. Later, no. 2, machine was taken out.

Of the three existing fourdrinier machines, nos. 3 and 4 were Harper fourdriniers, the other a standard fourdrinier. A greater variety of grades was desired and to do this, it was decided to rebuild no. 4 paper machine, to increase the speed from 300 to 500 f.p.m., increase the tonnage by about 25% and allow heavier grades to be made.

This meant rebuilding the complete machine to include a new fourdrinier, couch, additional driers, reel, winder, and complete drive. To do this a wall at the wet end had to be removed and the foundations under the machine had to be rebuilt. This also required additional capacity in the substation involving a 1500 kva. transformer addition.

The required changes to the piping and stock preparation equipment were done by mill personnel and all the existing piping was replaced with stainless steel. In addition, primary and secondary Bauer Centri·Cleaners were installed and additional refining equipment added.



Bauer Centri·Cleaners installed at the Lachute mill of J. C. Wilson, Ltd.

The existing flowbox was relocated to its new position and the slice was rebuilt by the mill from a vertical slice to a sloping type.

The old Harper fourdrinier which had a wire 94 in. by 40 ft. was replaced by a Dominion nonremovable shaking fourdrinier (with a shaking breast roll and table roll section). The wire is 94 in. by 70.5 ft. long and is level from the breast roll to the couch. The shake mechanism is a Dominion standard adjustable shake unit. This fourdrinier is equipped with the new Dominion patented (1958) torsion bar suspension. The rails and breast roll are mounted on torsion bars instead of the older type leaf springs. This arrangement gives better control of the shake with a worth-while saving in power.

The rolls include: bronze breast rolled 20 in. diam.; 32 brass table rolls of $4^1/_2$ in. diam.; four brass wire rolls of 8 in. diam.

A forming board with drilled Celeron top was also supplied.

There are six stainless steel suction boxes 6 in. wide with 4 in. openings. Each box has an end-of-grain maple cover.

The saveall trays are of stainless steel and extend under the complete table roll section. These savealls were designed for easy removal at a wire change.

The table roll rails are built up from steel plate and structural sections, stainless steel covered.

The couch is a Dominion open ended direct-driven cantilever type with a single suction box $6^7/_8$ in. wide. There is an internal shower for lubrication of the sealing strip and a tail blower for sheet transfer.

The draw roll located after the couch is a 4 in. aluminum roll mounted in antifriction bearings.

The original press section, which includes a plain straight through first press, and a plain reverse second press, was relocated toward the wet end to allow room for additional driers.

Four additional 48 in. diameter driers were installed at the wet end of the drier section, bringing the total number of driers to 17.

The two existing calender stacks were reconditioned by Dominion Engineering in their shops with new ways, and the bearings housings refitted to the ways.

A new Dominion Pope-type reel was installed, designed to reel a maximum paper roll 60 in. in diameter to permit two 42 in. diam. rolls to be made on the winder.

The drum is of 42 in. diam., arranged for water cooling.

Three $12^5/_8$ in. diam. reel bars were also supplied.

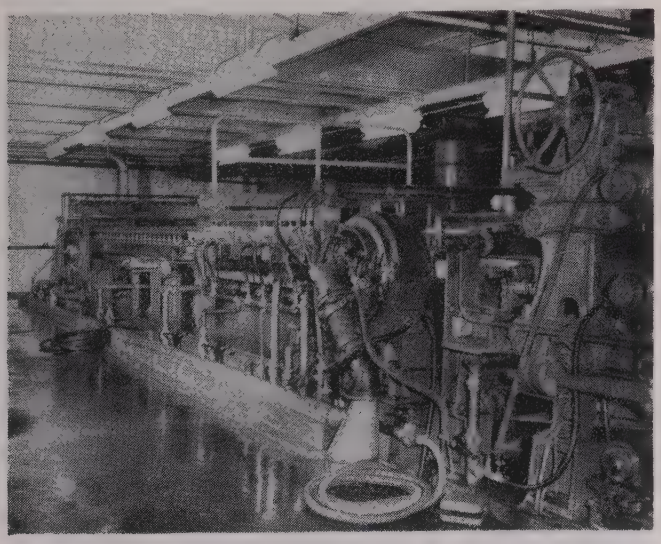
The Dominion unwind stand has cross and lengthwise adjustment, and is provided with a water cooled mechanical brake. It will accept the 60 in. diam. rools made on the reel.

The Black Clawson winder arranged with score cut slitters, is driven with a d.c. motor.

The drive, as supplied by Dominion, consists of seven spiral bevel drive units, one each for the couch, first and second presses, drier section, first and second calender stacks, and the reel.

Each drive unit is complete with a spiral bevel gear unit, a unit cone pulley, air-actuated Wichita clutch, electrically operated belt shifter and bed plate. The necessary controls were supplied for operation of the clutches to allow controlled starting of all sections and controlled inching of the drier section.

The line shaft, mounted in antifriction bearings, with its pulleys, is located in the basement, and is driven by a d.c. motor.



Dominion wet end of the machine installed at J. C. Wilson, Ltd.

Canadian International

A corrugated container plant to cost $400,000 will be constructed immediately in the St. John's, Nfld., area by Hygrade Containers Ltd., a subsidiary of Canadian International Paper Co., it was disclosed by the Hon. Joseph R. Smallwood, premier of Newfoundland, and by P. A. Sargent, vice-president of Canadian International Paper Co.

The plant will be in operation by early summer and will provide permanent employment for about 30 people, it was announced. It will be a modern, one-story building.

The output of the new plant will be distributed by N. C. Hutton Ltd. of St. John's. This firm, a prominent supplier of packaging materials, has been Newfoundland's sales representative for Hygrade Containers for a number of years. Its president, Noel Hutton, played a leading role in recent discussions which resulted in the company's decision.

The announcement followed a conference between Mr. Sargent and Premier Smallwood. Also present at the conference were Gordon F. Pushie, Newfoundland's director general of economic development, and J. T. Woolley, general manager of Hygrade Containers.

Business has now grown in Newfoundland to the point at which a manufacturing plant to provide faster service for local customers and to eliminate unnecessary warehousing on their part has become imperative, Mr. Sargent stated.

The Hygrade Containers plant will be in operation by early July. It will be the first plant manufacturing corrugated containers in the province.

Hygrade Containers Limited now operates corrugated container plants at London, Ont.; at Rexdale, near Toronto, Ont.; and at Pointe-aux-Trembles, just outside Montreal, P. Q.

Champion

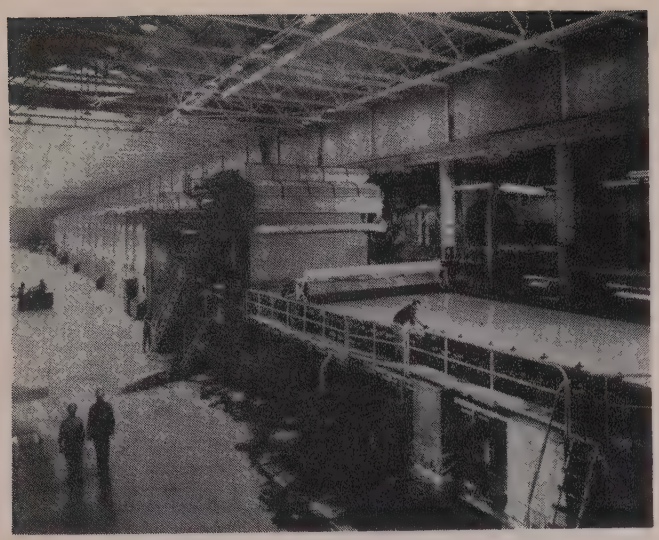
As an historic bell rang, an overhead crane pulled back curtains May 1 to reveal the world's largest fine papermaking machine.

Over 400 ft. long and rated as capable of producing paper in a strip 20 ft. wide at the rate of 2000 f.p.m., the machine was formally commissioned at ceremonies in Canton, N. C.

Attending the ceremonies were North Carolina's Gov. Luther H. Hodges, officials of the Champion Paper and Fibre Co., for which the ultramodern machine was specially designed, and invited guests from all over the country.

At a luncheon preceding the unveiling, Governor Hodges said the new machine, a multimillion dollar investment which alone can boost Champion paper production another 90,000 to 100,000 tons of paper annually, is a "significant step forward in realizing the industrial potential of the state."

He noted that no. 20 machine has meant employment of over 200 new people at Champion's Carolina Division and will swell the firm's Canton payroll another million dollars annually to $18 million. (Champion also has manufacturing divisions in Hamilton, O., and Pasadena, Tex.)



The size of no. 20 machine is shown by this photo in which the men at the machine's far end can barely be seen. A steam turbine provides the machine's driving power. This turbine develops 2400 hp., equivalent to 1800 kw.— or enough to supply the average electrical requirements of a town of 5000 people

The bell, which was rung four times by Champion's Board Chairman Reuben B. Robertson to "commission" the machine was first used in 1906 to call workers to their jobs when the firm's plant at Canton was first being erected and before there was any steam in boilers to blow a whistle.

Pointing out the technological progress since then, Mr. Robertson said, "I came to Canton in 1906 on what was supposed to be a 50-day assignment, which lasted more than 50 years. When I arrived here, the grading for the mill site was still in progress and the superintendent pointed with pride to the fact that they were using the very latest improvement in dirt moving machinery, the horse-drawn, wheeled Fresno scraper, which displaced the old fashioned pan scraper and moved about four times as much dirt to the trip. Quite a difference from the way things are done today."

No. 20 machine, which has been in the planning and construction stage since 1953, also called for construction of an enormous building to house it and for additional structures for preparing the stock mixture which flows through the machine to ultimately become paper and for "finishing" the newly produced paper for customer use.

Reuben B. Robertson, Jr., Champion's president, in brief remarks noted that the new machine "symbolizes how a free society builds for present and future growth."

No. 20 machine is the world's largest for the manufacture of fine papers. The machine is capable of operating at a speed of 2000 f.p.m., producing more than a mile of paper every three minutes in a sheet 230-in. wide.

Other facts concerning the no. 20 and the attendent construction are:

Length of machine and winder, 420 ft.
Width of fourdrinier wire, 246 in.
Height of machine, 27.5 ft. at presses and 25.5 ft. at calenders.
Driers. Hooded, of latest design, incorporating features that assure high efficiency and uniformity of drying of fast moving sheets of paper. The hood also provides improved working conditions in the areas surrounding the machine.
Calenders. The two calender stacks are $25^1/_2$ ft. in height and are equipped with new pneumatic nip-relieving devices.
Winder. A double drum Beloit Model L. Maximum speed 6000 f.p.m. Driven with 250 hp. d.c. motor.
Two Overhead Cranes. One 40-ton capacity, one 25-ton capacity. Have two 20-ton hooks and three $12^1/_2$-ton hooks.
Machine Building. Two stories high; 515 ft. long; 124,000 sq. ft. floor space.
Stock Preparation Building. Four stories high, 50,000 sq. ft. of floor space.
Thickeners. Three; Champion Paper and Fibre Co. design and manufacture, 6-ft. diameter drum and 12 ft. in length. Capacity 200 tons per day.
Storage Chests. Four; pine chest has 16-ton capacity; hardwood, broke and machine chests have 12-ton capacity.
Jordans. Thirteen; Emerson 4-K, 400 hp. and 400 r.p.m.
Hydrapulpers. Three; 20-ft. diameter Black & Clawson; capacity 180 tons per day on 25-min. cycle.
Addition to Paper Finishing Area. 43,000 sq. ft. of floor space. (Includes main mill entrance with covered portico.)
No. 20 machine was designed by Champion engineers and manufactured by Beloit Iron Works, Beloit, Wis.

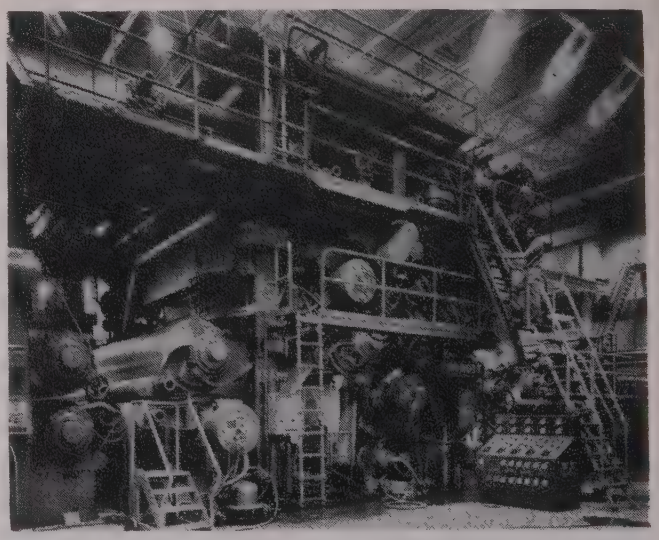
Stock Preparation

The stock from the pulp mill storage tanks is received into the stock preparation system over three 6 by 12 saveall thickeners. The thickened stock flows to the hydrapulpers for mixing of the supplemental furnishes and on directly to the storage chest ahead of the jordans. Ten jordans refine the stock prior to discharge to the tile lined machine stock chest. This equipment is all contained in the stock preparation building, a four-story, brick and concrete structure. This building contains all of the above equipment and its necessary accessories, controls, and motor control centers. In addition, it provides storage space for wet lap pulp awaiting introduction to the system via the hydrapulpers.

The stock from the machine chest is given the final refining in the tickler jordans before entering the basis weight head tank. From the basis weight head tank, stock is introduced to the primary Centri-Cleaner system through a Worthington 20 LA type pump with 1250 hp. drive unit. Accepted stock from the 29 primary cleaners passes through a bank of selectifier screens and into the pressure headbox.

Paper Machine

No. 20 paper machine is a 246-in. fourdrinier machine, right-hand drive, designed to produce at speeds of 2000 f.p.m. The prime mover tubine driving the differential type mechanical line shaft connects with the wire turn roll, first and third press rolls, smoothing press, four drier sections, size press, calenders, and the reel. In-line generators provide electric power for the drives on the couch wire return rolls, second press wringer rolls, pickup roll, felt turning suction rolls, top size press roll, and various tension rolls. Each differential drive has P.I.V. speed control with air operated clutch connection between the differential and the transfer gear cases on the in-drive. Electric inching drives are provided for the calenders and reel.



The press roll section, built vertically instead of horizontally in order to conserve space, is 27 1/2 ft. high. These presses, exerting tremendous force, squeeze out additional water from the newly formed web of paper as it leaves the fourdrinier screen section

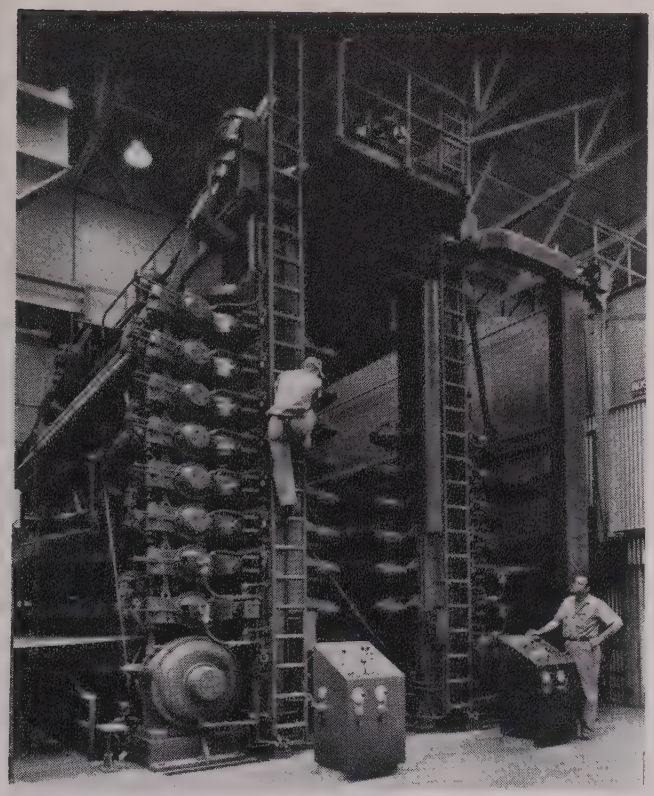
The steam turbine is a 2400 hp. multivane, multistage noncondensing, 400 p.s.i. inlet unit. This unit has a 10:1 speed range.

The headbox is of the latest air pressure design, incorporating a multiple pipe dual impingement distributor. The box contains five variable speed rectifier rolls and is equipped with an adjustable straight-lip slice. The level in the headbox is automatically controlled by recirculation of stock to the primary Centri Cleaner pump. Pressure is provided by a Nash L-3 compressor.

Stock from the headbox is delivered onto a 246-in. fourdrinier of the removable, shaking type. Major components of the fourdrinier are as follows: 36 in. diam. breast roll with air motor lowering device; twenty-seven 13 in. diam. table rolls; seven stainless steel suction boxes with provision for oscillation; 30 in. diam. dandy roll; seven wire return rolls; two position wire tension rolls; and automatic wire tension control mechanism. The 42 in. diam. couch has both high and low vacuum suction boxes. The fourdrinier saveall pans return water to a wet-type wire pit. This pit is designed to remove air, and has foam removal skimming weirs. This and the couch and press pits are of tile construction.

The suction pickup press is of the inclined three-roll type, followed by a straight-through third suction press and smoothing press. The first three presses are provided with conventional lubricating showers, suction box, felt conditioner, and necessary felt cleaning equipment required for operation of this type of installation. The top of the press section towers 27 ft. above the operating floor.

The paper driers, six Feeney driers, and eight return felt driers are divided into four sections with the size press located between the third and fourth sections. The driers are all 5 ft. in diam., designed for 125 p.s.i. steam pressure. The condensate removal system is of the forced vapor circulation type. The drier hood sections are total enclosed design made up of aluminum insulated and corrugated panel sections. These sections enclose the drier sections up to the size press, with a separate hood section for the remainder of the driers. Each hood section is furnished with heated make-up air into the basement with provisions for recirculation and exhaust temperature control to eliminate heat loss from the system.



The calender stacks, where the paper is "ironed smooth" by pressure rolls, are 25 1/2 ft. high. They are equipped with a pneumatic device to spread the heavy rolls apart

The two open-side calender stacks are provided with nip-relieving systems, electrically operated roll lifts, oscillating doctors, and air cooling systems. Both stacks have 36 in. diameter king rolls. The first stack contains seven rolls, while the second is a nine-roll stack.

The reel is a heavy duty constant speed drum type with primary and secondary air loaded reel arms for continuous reeling at high speed. The 36 in. diam. drum is designed to handle 88 in. diam. log rolls. The reel is provided with a full width braking system.

The heavy duty double drum winder unit is designed for speeds of 6000 f.p.m. Each drum is driven by a 250 hp. d.c. motor. Oscillating unwind stand, individually driven sheet slitters, and automatic shaft removal are provided on the unit. Complete conveyors, necessary transfers, roll wrapping, and weighing systems have been installed with the winder.

The machine is provided with a broke system consisting of three machine width repulpers of stainless steel hopper construction. Each of these repulpers is provided with automatic consistency control and agitator equipment.

In conjunction with the machine building, a new shipping building and finishing room area, including office space, have been provided. Storage area and loading facilities with space for handling ten railroad cars inside the building were a part of this project.

Miscellaneous and Auxiliary Facilities

Electric power connections from the mill system, as well as a block load tie with the Carolina Power and Light Co., were installed for this machine and auxiliary equipment.

In parallel with this project, a new mill clay handling system was installed.

A 40 ft. diam. by 30 ft. high storage tank was installed as the heart of a fiber recovery system to handle any chest dumps, thickener overflows, or excess broke during the periods of machine operations.

MOHAWK

An expansion and modernization program at Mohawk Paper Mills, Inc., Cohoes, N. Y., has included installation of three new Langston paper machine winders, new reels, and the addition of new warehouse space.

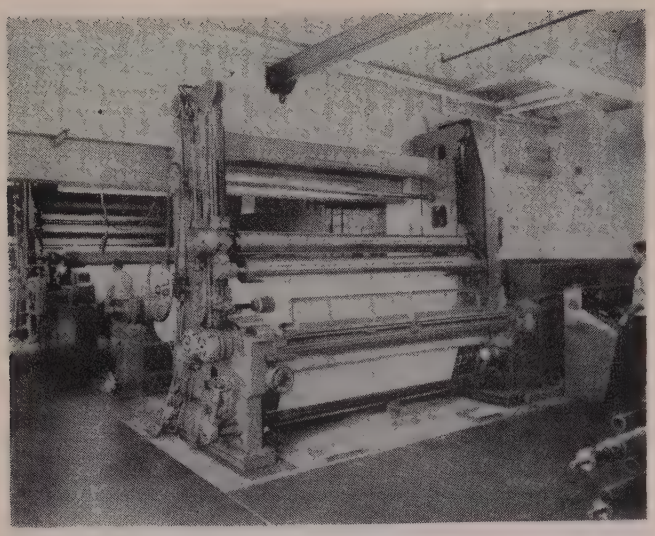
A nonintegrated producer of various texts, covers, vellums, and specialty papers, the company operates three fourdriniers, one at Cohoes about 10 miles north of Albany and two at Waterford several miles farther north, turning out about 24,000 tons annually.

Expansion and modernization is very much in evidence at the company's plants. A new warehouse at the Cohoes Division provides 48,000 sq. ft. of storage space for finished paper. A new warehouse at the Waterford Division is of equal size, and at Waterford a new 14,000 sq.-ft. building has just been completed where two cutters will soon be installed.

At Cohoes, the 110-in. Sandy Hill paper machine has been fitted with a new Rolla-Reel, and plans have been made to install a new drive, according to T. Charles Nixon, Cohoes mill manager.

At Waterford, the two 72-in. Sandy Hill fourdriniers have been improved recently with Pope-type reels.

But, outstanding at both mills, according to company officials, has been the new installation of three paper machine slitter and winders built by Samuel M. Langston Co.



Langston winder on 110-in. paper machine at Cohoes, N. Y., mill of Mohawk Paper Mills, Inc., slits and rewinds fine grades of text, vellum, cover and ledger, ranging from 13-lb. (17 by 22) basis to 175-lb. (25 by 38) basis

The Langston winder at Cohoes is a 112-in. machine. According to production manager Myron Flotow, "It is doing an excellent job of rewinding tight, compact, evenly wound rolls."

Says Flotow: "Our Cohoes production ranges from 13-lb. (17 by 22) basis to 175-lb. (25 by 38) basis. We're rewinding into rolls that vary from 9 in. wide by 40 in. in diameter to two 50 in. wide rolls by 35 in. in diameter.

"The Langston winder keeps up with production, makes nice clean cuts, is easy for the men to learn to run and operate, and easy to thread through. The slitters are handy to get at for setup. We're happy about the way this new winder is performing."

The production at Cohoes includes the finer grades of texts, covers, vellums, and ledgers, mostly white. After they are slit and rewound, the rolls are taken to either of two, 110-in. wide rotary duplex cutters where they are sheeted to specifications.

The two paper machines at Waterford, in addition to poster and construction papers, produce specialty papers for box facings, linings, and covers, such as Mohawk's Navajo cover which comes in 12 colors. Here also are made feltmark papers such as Artemis, Cortlea, and Diana.

Both machines are running a 66-in. wide sheet ranging from 30-lb. to 150-lb. (24 by 38) basis weight. Langston 72-in. wide slitter and winders slit and rewind the sheet to specifications.

"The Langston winders have made a big improvement in our rolls," says Waterford plant superintendent William J. Roberts, Sr. "The rolls are harder and they are wound off evener than we have ever been able to accomplish before," he adds.

Otto Zieball, assistant manager, says: "The shear-cut slitters are very good. Their action is sharp and clean. They are trouble free. We really approve of the fixed-shear angle feature of the slitters. There's practically no chance of improper setup or their getting out of line. And the Mt. Hope rolls are important. They give good separation."

Each Langston winder utilizes a General Electric variable speed drive. Each is a precision built machine, engineered to produce the finest quality slitting and winding.

All rotating rolls are dynamically balanced for 500 f.p.m. in excess of contract speeds—a Langston standard feature. Each machine is equipped with Langston's time-proved clean cut, shear cut slitters. Other Langston standards on the equipment include console-mounted and operator controlled rider roll torque drive and rider roll hold-down to tightly secure the sheet to the core and achieve a good hard roll center.

The winder at Cohoes in addition is equipped with roll ejector and Langston patented rider roll density controller. Controls for these features are console-mounted for operator control.

C-Z

Crown Zellerbach Corp. is investigating the possibility of establishing a newsprint mill in the vicinity of Denver to supply its newspaper customers in the Mountain States region.

The company will shortly undertake intensive studies of the various economic factors involved in the project, including timber availability, labor, water and power resources, and possible millsites. The millsite phase of the study will include the upper Colorado River Valley.

St. Regis

Construction of a million dollar corrugated box plant, first major manufacturing start in the new Bridgeview, Ill., Industrial District, was announced by Indiana Harbor Belt Railroad, Village of Bridgeview, and Ajax Box Division of St. Regis Paper Co.

The 165,000 sq. ft. Ajax Box plant was designed by Ralph Stoetzel, architect and engineer, 100 W. Monroe St., Chicago, and is being built by Miller-Davis Co., 38 S. Dearborn St., Chicago. Construction started in March with a completion target of Oct. 1, 1959.

Ajax Box, Chicago manufacturer of corrugated containers and specialty products since 1928, now is located at 2210 S. Union Ave., in the path of the new southwest expressway.

Details of the new plant and industrial district, 68th to 95th St. west of Harlem on Chicago's southwest perimeter, were revealed by Robert S. Prentice, Ajax Box general manager; Henry W. Coffman, manager of industrial development for New York Central Railroad; John Oremus, Bridgeview village president, and architect Stoetzel.

The Bridgeview Industrial District, a 300-acre tract, is being developed by New York Central through its Indiana Harbor Belt affiliate in conjunction with Bridgeview. The village will provide streets, sewer systems, water facilities, and is building a new water plant for the community. The district also has all other major utilities required by industry.

The Ajax Box plant is the second new container manufacturing unit announced for the midwest in as many weeks by the St. Regis Container Division. Another million dollar, 200,000 sq. ft. plant is being built at Dubuque, Ia. The company operates some 30 corrugated, folding, and setup box plants in its container division.

St. Regis is one of the country's largest integrated paper companies, with major timberland holdings; pulp & paper mills; multiwall bag, container and packaging equipment plants; printing and specialty paper plants; extruded and laminated plastics plants, and building products plants located in most sections of the United States, as well as extensive international operations.

Prentice said that Ajax Box purchased 16 acres within the Bridgeview Industrial District for the plant and local offices. The plant, a one-story steel and brick building with two-story adjoining offices, will have a designed capacity of 35,000 tons of corrugated product annually. A new 85-in. corrugator will be installed initially. Addition of other modern pieces of converting equipment is contemplated. The plant is designed and will be constructed so that it can be easily expanded for additional production lines and products.

St. Regis Paper Co. has filed with the Securities and Exchange Commission a registration statement relative to the issuance of shares of St. Regis common stock to be offered in exchange for the 110,000 outstanding shares of common stock of Chemical Packaging Corp. of Savannah, Ga.

The proposed offer would provide that if the exchange offer is declared effective, a total of not exceeding 20,000 shares of St. Regis common stock would be issued on the basis of one share of St. Regis stock for $5^1/_2$ shares of Chemical Packaging stock.

Chemical Packaging manufactures and sells multiwall paper bags primarily for packaging chemical fertilizer to manufacturers in the southeastern area of the United States. Chemical's two bag plants are located at Savannah, Ga., and Louisville, Ky. Sales of Chemical Packaging in 1958 were in excess of $3 million.

Acquisition by St. Regis of the shares of Chemical common stock will provide St. Regis with additional bag manufacturing facilities for serving customers in the southeastern area of the United States and extend the multiwall bag business of St. Regis into an area which includes manufacturers of chemical fertilizer not presently served by St. Regis.

O-I

The Los Angeles plant of National Container Corp. of California, a subsidiary of Owens-Illinois Glass Co., is undergoing a modernization and expansion program that will more than double the plant's present size.

George J. Schneider, president and general manager of National Container, said the 169,000 sq. ft. of floor space being added to the plant will also double the plant's production capacity.

Substantial quantities of new equipment, including a new 85-in. corrugator, will be added to the plant's present facilities. The existing corrugator and other production machinery will be realigned to permit more efficient flow of materials and product.

Construction, now underway, should be completed this fall, Mr. Schneider said.

The plant addition will be of tilt-up concrete panel construction with steel roof framing and Owens-Corning fiberglas roof.

The program calls for addition of new office space, a new receiving dock for rail shipments and a new shipping dock for the company's trucks, and a new parking lot to accommodate 130 cars.

Other new construction will include addition of one new boiler, installation of new glue-mixing equipment, and new scrap collection and baling facilities. A new water storage tank for fire protection will be built, and locker rooms for employees.

The company acquired four additional acres in connection with the plant expansion program.

General contractor is Donald F. Shaw of Los Angeles.

Hollingsworth & Whitney

A new type of antiskid paper which gives maximum protection against slippage of multiwall bags during transit or storage has been announced by the Hollingsworth & Whitney Division of Scott Paper Co.

When used for the exterior ply of multiwall bags, the new H&W Gripper-Kraft stock provides a gripping surface proved far more tenacious than rough finish kraft or other conventional nonslip coated papers.

H&W engineers report the new Gripper-Kraft is applicable to a wide variety of packaging uses in the chemical, food, fertilizer, mining, cement, and other industries where damage to bags during shipment or storage in warehouses is a major problem.

The new stock is the product of a manufacturing process developed by Hollingsworth & Whitney. In this process, the surface of the regular finish H&W multiwall paper is treated with a special antiskid chemical right on the paper machine.

This is a radical departure from previously used methods. Conventional nonslip coatings are sprayed on the paper surface in a separate process after manufacture—resulting in added expense to the manufacturer or converter.

Prior to the development of its new nonskid paper, H&W made a rough finish kraft to accomplish the same nonslip purpose in its multiwall grades. The new Gripper-Kraft not only has greater efficiency than the rough kraft, but also avoids certain inherent disadvantages of the rough finish paper.

Less ink is required to print on the Gripper-Kraft, because its smoother surface presents less area to be covered than the rough kraft.

To determine the relative nonskid qualities, H&W had a series of tests conducted, pitting the new Gripper-Kraft against the rough finish stock.

Placed on an inclined plane, bags made of Gripper-Kraft showed 15% less slippage than bags made of the rough finish kraft. In another test, 100-lb. bags of material were pushed from the top of a pile of bags. It took 20% more force to dislodge the Gripper-Kraft bag.

It was also found that the new antiskid paper is superior to rough finish papers on metal and wood chutes. Because of its smoother surface, more of the finish is in contact with the surface of the chute—in contrast with the irregular surface of the rougher paper. As a result, there is more resistance, slowing the slide of the bag. With a slower slide, there is less impact if the bags should bump each other—and therefore less tearing and breakage.

The manufacturer points out that, in obtaining the antiskid qualities, the strength of the paper is in no way sacrificed. Gripper-Kraft meets all government specifications and has a low porosity of 10 sec. maximum. The new stock does not attract lint or dirt.

I.P.C.

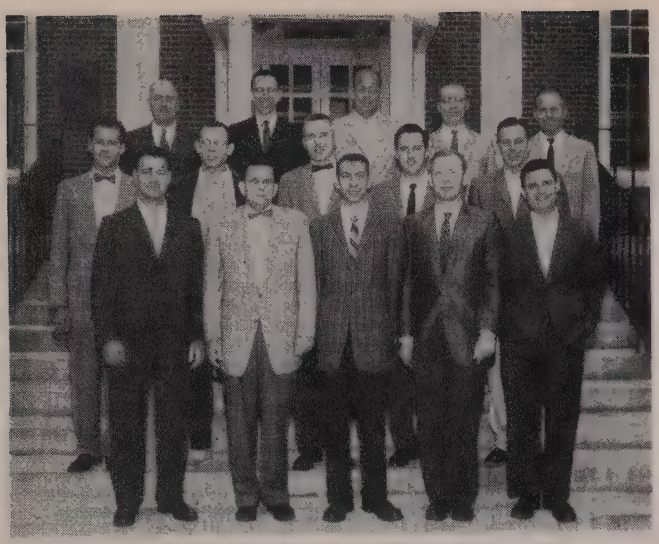
Research staff and students at The Institute of Paper Chemistry will be able to work on new types of problems and to devote more time to actual experimentation through the use of an IBM 610 automatic computer recently installed at the institute.

The 610, a baby brother to the giant automatic computers used in military and space programs, is designed to free the individual scientist and engineer from routine computations. Occupying about as much space as a standard kitchen range, the 610 automatically carries out the average scientific calculation at 10 times the speed of a skilled technician using a desk calculator. In complex types of statistical analyses, it can turn out more work in an hour than the calculator operator can in a week.

In announcing its acquisition, Roy P. Whitney, vice-president and dean of the institute, stated that by means of the computer the institute could develop avenues of research formerly blocked by the need of time-consuming technical calculations. The use of a computer, he also noted, often leads to a new slant on a program pursued for some time, because the research worker gains a heightened appreciation of the mathematics involved.

Operation of the 610 computer at The Institute of Paper Chemistry is a cooperative enterprise between the institute and the International Business Machines Corp. As a part of its "University Program" the IBM Corp. is assuming a portion of the cost of placing 30 units of this newly developed type of computer at various collegiate institutions throughout the nation.

Because this machine has been designed for ready use by any engineer or researcher in science, the institute will not employ a professional computer staff, such as is required in larger installations. Staff members and students have been participating in training sessions in the use of the equipment. Edward F. Thode of the Chemical Engineering Department has been designated to coordinate the various computer-related activities.



Completing the 2-week paper summer course at Lowell Technological Institute sponsored by the Boston Paper Trade Association and the New England Paper Merchants Association were those shown, l. to r. First row: Maurice Furchgott and Joseph H. Beazley, Montag Bros. Inc.; Francis J. Scavito, Crown Chemical Corp.; Prof. Norwood H. Keeney and Charles J. Higgins of the L.T.I. instructional staff. Second row: Peter R. Fritzsching, Chemical Research Central Research Laboratories; Charles A. Simberg, Connecticut Container Corp.; John B. Allen, The Rourke-Eno Paper Co.; James W. Anderson, Whitney-Anderson Paper Co.; J. Regan, National Aniline Division, Allied Chemical Corp. Third row: Prof. Edward M. Engel of LTI; Robert L. McDowell, American Viscose Co.; David E. York, Crown Chemical Corp.; Richard Cowles, The Rourke-Eno Paper Co.; Prof. John Lewis, head of LTI's paper engineering department

L.T.I.

Eleven men completed the paper summer course held at Lowell Technological Institute June 15 to 26 under the sponsorship of the Boston Paper Trade Association and the New England Paper Merchants Association. Enrolled were the following:

James W. Anderson, Whitney-Anderson Paper Co., Springfield; John B. Allen and Richard Cowles, The Rourke-Eno Paper Co., Hartford, Conn.; Joseph H. Beazley and Maurice Furchgott, Montag Bros. Inc., Atlanta, Ga.; Peter R. Fritzsching, Chemical Research Central Research Laboratories, Murrayhill, N. J.; Robert L. McDowell, American Viscose Co., Marcus Hook, Pa.; J. Regan, National Aniline Division, Allied Chemical Corp., Buffalo, N. Y.; Francis J. Scavito and David E. York, Crown Chemical Corp., Providence, R. I.; Charles A. Simberg, Connecticut Container Corp., Newton.

Conducting the course were members of LTI's paper engineering department, Prof. John Lewis, department head, Prof. Norwood H. Keeney, Prof. Edward M. Engel, and Charles J. Higgins. Various seminars were conducted by guest lecturers from industry, among them the following:

Russell Hamilton, mill manager, and Harold Logan, chief chemist, St. Regis Paper Co., East Pepperell, who discussed kraft papers; A. M. Altieri, chief chemist, and John Wendell, Tileston & Hollingsworth Co., Hyde Park, who spoke on fine papers; Warren Bennett, comptroller, Oxford Paper Co., Lawrence, whose program dealt with costs; William Bell, Lee Segouin, and James Bowler, Courier-Citizen Co. and Harold Cowley, International Printing Ink Division, Interchemical Corp., printing seminar; Eugene O. Hansen, Brown Co., Boston, speaking on paper sales from the mill angle.

Field trips were made to Oxford Paper Co., Lawrence; A & P Box Co. and the Associated Folding Box Co., Lawrence; Nashua Corp., Nashua, N. H.; Courier-Citizen Co., Lowell; St. Regis Paper Co. and Bemis Bros. Bag Co., both in East Pepperell. A graduation banquet with presentation of souvenir diplomas climaxed the course.

Lectures and demonstrations were presented in the following areas:

Pulp technology; stock preparation; beater additives; sheet formation; beater-pulp testing; beating theory; practical tests; machine tests; paper classification; beater tests; physical tests; methods of evaluating results; papermaking arithmetic; literature and statistics; printing processes; printing papers and inks; coated papers; beater additives laboratory; fine papers; kraft papers; specialty fine papers; tapes, labels, films, foils; reinforced and waterproof papers; and folding cartons and corrugated containers.

Columbia

Specialized training in economic writing and reporting will start this fall at Columbia University's Graduate School of Journalism, the school announced. The program will be financed by grants of The Clapp & Poliak Foundation, Inc., of New York.

The school will offer up to three Clapp & Poliak Foundation fellowships of $1500 each and up to three scholarships of $1000 each to entering students currently employed in the business communication field, or with training in economics and aptitude for journalism. The recipients will be admitted as regular students in the school's one-year graduate program and will receive the school's basic journalistic training. In addition, they will devote time to special seminars, lectures, and studies guided jointly by the Journalism School and the Graduate School of Business.

The program will also include a conference and research project designed to investigate the state of economic reporting, to bring together leaders in the field for exchanges of views and recommendations, and to experiment with presentation of economic news.

The Clapp & Poliak Foundation's support of the program was made possible by Saul Poliak, a 1926 graduate of the School of Journalism, who is president and chairman of the board of the foundation. The program is tentatively planned to continue for ten years.

Pitt

The University of Pittsburgh has been selected by the Scott Paper Co. Foundation as a participating school in its scholarship program in colleges and universities across the country.

Italian Section members at a picnic held May 29 at the home of Donald Macaulay, president of Paper Quality Control Inc., Chappaqua, N. Y. Front: Mario Mariscotti, Daniele Conte, Tom Di Palma, and Allesandro Restagno. Rear: Mario Gislon, Marco Fruttero, Carlo Cerrina, Donald Macaulay, Carlo Ruspa, Guiseppe Villa, Piero Vanzetti, R. T. Bingham, and Anita Zani

Known as the Scott Foundation Awards, the scholarships are awarded to outstanding sophomore students with a significant interest in seeking a career in the field of business or industry. These awards are currently in effect at 20 colleges and universities.

The scholarships—one of which will be awarded annually for the next five years at Pitt—provide for $1000 a year for the junior and senior years, a supplemental grant of $1000 a year to the university, and an opportunity for summer employment with Scott Paper Co.

The first Scott Foundation Award at Pitt will be made to a student for use in the fall of 1959. The student will be selected in the near future.

Michigan

Restoration of half a million Michigan acres to productivity and increased timber yields on thousands of other acres is the first research goal announced May 1 by the newly incorporated Lower Michigan Pulpwood Research Association (LMPRA), headquartered at Filer City.

LMPRA—financed by five companies in Michigan's pulp and paper industry—has granted $6164 to The University of Michigan School of Natural Resources for the study. In all, LMPRA will provide $15,000 this year to colleges, universities, and public and private research organizations for advancement of forest research and public information in scientific forestry.

Heading the research group are Profs. Samuel Graham and Stephen Spurr of the School's Department of Forestry. Graham directed the U-M's Phoenix Atomic Research Project aspen study whose results have had far reaching effects in wildlife and forest management.

Project One is entitled: "Reestablishment of Aspen Stands on Sites Where Forest Conditions Are Economically Suitable."

"Aspen pulpwood, harvested from trees once considered worthless weeds, is now a multimillion dollar annual forest crop in Michigan's Lower Peninsula," said LMPRA President Roman Suess of Otsego, who announced the grant to the U-M.

"Fast-growing and with excellent papermaking qualities, the aspen—commonly called popple—has been the economic life-blood of many northern communities. Lush aspen regrowth following logging has been the salvation of our deer herds in many areas," he continued.

Surveys have shown that more than one-half million acres alone in the Lower Peninsula could be growing aspen, Mr. Suess said. "Instead they are virtually devoid of valuable trees. We aim to find a remedy."

Mr. Suess is also president of the Otsego Falls Paper Mills. Four other lower Michigan pulpwood companies also are represented by LMPRA officers: Abitibi Corp., Alpena; American Box Board Co., Filer City; Scott Paper Co., Detroit; and S. D. Warren Co., Muskegon.

N. C. State

The Carolina Lumber and Building Supply Dealers Association has established a $500 scholarship award in the School of Forestry at North Carolina State College.

In reporting this today, association officials said the award will be known as the E. M. Garner Scholarship in honor of E. M. Garner of Charlotte, the association's executive secretary.

The scholarship recipient will be chosen annually from among applicants planning to enroll in the college's School of Forestry.

Members of the Carolina Lumber and Building Supply Association have been working with the School of Forestry and for several years have sponsored a four-year college program called Lumber and Building Products Merchandising.

E. Reid Hinson, Jr., of Charlotte (dressed in ROTC uniform), junior in pulp and paper technology at North Carolina State College, receives the Fred C. Boyce Student Award, presented by the Paper Industry Management Association. Presenting the award on behalf of the association is Prof. C. E. Libby (fourth from left), head of the pulp and paper technology curriculum at North Carolina State College. Looking on are Hinson's professors and classmates

Dorr-Oliver

Dorr-Oliver Inc. announces the availability of five new equipment bulletins bound in a single catalog entitled, "Dorr-Oliver Pumps for Hot or Cold Corrosive and Slurry Service." Containing 22 pages, the catalog describes the complete D-O pump line for chemical process and allied industries. Included are line and wash drawings, photographs, specification and performance data, and parts lists. Copies are available from Dorr-Oliver, Inc., Havemeyer Lane, Stanford, Conn.

Thwing-Albert

Thwing-Albert Instrument Co. announces the publication of Bulletin 1051-TA which describes the T-A-C Tester and its operation, application, and advantages as a laboratory means of quickly and easily checking the suitability of an adhesive to an adherend under simulated production-use conditions.

Copies of Bulletin 1051-TA may be obtained from the Thwing-Albert Instrument Co. at Penn St. & Pulaski Ave., Philadelphia 44, Pa.

Hercules

The commercial introduction of a new chemical, with unusual properties that suggest its use in a wide variety of applications, has been announced by Hercules Powder Co.

This new product, Natrosol 250, is a water-soluble stabilizer and thickener. Extensive development work suggests its application in water-based emulsion paints, preparation of polyvinyl acetate emulsions, textile warp and finish sizes, paper coatings and paper sizings, ceramics, inks, and latex emulsions for adhesives and coatings.

Natrosol is a nonionic water-soluble hydroxyethyl ether of cellulose, available in four viscosity types. One of its unique properties is that one viscosity type is the highest available for this type of chemical, an advantage which suggests Natrosol as the most efficient thickener among nonionic cellulosics.

Lawson

William Schaaf has been transferred from the New York office of The Lawson Co., Division of Miehle-Goss-Dexter, Inc., to the company's Chicago headquarters, and has been

William Schaaf, The Lawson Co.

appointed to the newly created post of manager of technical services.

In announcing the move, Carlton Mellick, Lawson's director of sales, said that it was prompted by a desire to be of maximum service to Lawson cutter, trimmer, and drill users throughout the country. In his new position, Mr. Schaaff's primary responsibility will be for the continuing technical training of Lawson and dealer service personnel. In addition, he will act as liaison between sales, manufacturing, and service within the company.

HOOKER

Hooker Chemical Corp., Niagara Falls, N. Y., announces the assignment of Canadian pat. 571,741, issued March 3, 1959, covering a process for production of high quality calcium hypochlorite bleach liquor automatically and continuously at rates of from 5000 to 15,000 gal. per day and higher. The process can be licensed for a nominal fee. United States patent application is now being processed.

The new system was developed exclusively by Hooker as a service to the pulp and paper industry. It is replacing the conventional batch operation which is wasteful of chemicals and requires operating labor. Three of the Hooker automatic systems have been operating for a year or more in Western Canada while two more are operating in the western United States. All are producing large quantities of bleach liquor on a continuous basis.

Indications from the units now operating show savings in chemicals amounting to some 2 to 3% over the old batch process. Significant labor savings are made and capital investment is greatly reduced as compared with the previous costly and space-consuming batch systems.

Several different types of reaction equipment have been used successfully in the operating system, such as rigid pipe coils, flexible hose coils, and draft tubes. Hooker points out that careful engineering is required to take advantage of optimum savings. This includes consideration of lime supply to the system which has proved to be a critical factor.

Detailed information on these calcium hypochlorite units and on the related automatic sodium hypochlorite units may be obtained by writing to Hooker Technical Service, either at Niagara Falls, N. Y., Tacoma, Wash., or at Hooker Chemicals Limited, North Vancouver, B. C.

Formation of the Western Chemical Division of Hooker Chemical Corp. has been announced by Thomas E. Moffitt, president of Hooker and of Hooker Chemicals Ltd., at company headquarters in Niagara Falls, N. Y. The new division will consist of Hooker's plants at Tacoma and Spokane, Wash., the plant of Hooker's Canadian subsidiary, Hooker Chemicals Ltd. at North Vancouver, B. C., a bulk storage terminal at Wilmington, Calif., and the sales organizations with offices at Tacoma, Los Angeles, and North Vancouver. This marks the final step of divisionalizing the 12 plants of Hooker into the Eastern Chemical, Western Chemical, Phosphorus, and Durez Plastics Divisions.

The Spokane plant, which manufactures and supplies wood flour to the western fiberboard industry, now becomes part of the Western Chemical Division, rather than of the Durex Plastics Division as formerly.

Horace W. Hooker, formerly western sales manager, is appointed sales manager, Western Chemical Division, and George A. Gentes, who has been works manager of the Tacoma plant, is named production manager of the division, Mr. Moffitt also announced. Mr. Hooker will report to Robert E. Wilkin, a senior vice-president, while Mr. Gentes is responsible to Mr. Moffitt.

BOLTON

Following years of technical development and field testing, John W. Bolton & Sons, Inc. of Lawrence, Mass., announces its Boltobar one-piece jordan shell filling.

Now in full production, Boltobar fillings are made to close tolerances in sizes to fit standard jordans of any make or size.

The new one-piece filling reduces maintenance costs.

Boltobar one-piece jordan shell filling

Fifteen minutes is all that is generally required for installation, making a substantial saving in down time.

Boltobar shell fillings are secured through development of a unique internal fastening structure. As in all jordan fillings made by Bolton, knives are fabricated by the exclusive Microlyzing process. Specially treated wood fillers maintain their shape during operation, yet swell when wet to make an extremely tight fit. As a result, knives are securely anchored during operation.

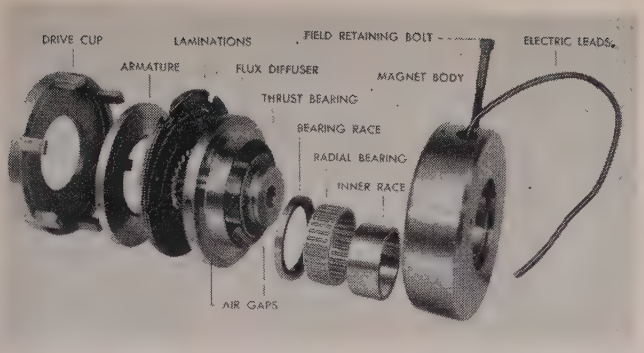
I-T-E

A novel stationary-field magnetic clutch, which virtually eliminates the need for maintenance in machine transmissions, has been added to the line of Electro Clutches made by I-T-E Circuit Breaker Co., Philadelphia, Pa.

The new clutches also permit reductions in the size of machine tool transmissions because they provide more torque for their size than any other units commercially available.

These clutches can be buried in a machine drive box or transmission and forgotten because there are no air gap adjustments to be made, no slip rings and brushes to be cleaned, adjusted, or replaced.

The new design, incorporating a closed flux path through hardened steel laminations, eliminates the need for adjusting air gaps. Use of a stationary magnetic field—coil windings



Assembly details of new clutch are shown in exploded view of all major component parts

are on the stator instead of on the rotor—eliminates slip rings and brushes.

These new clutches open up many new possibilities for use in drive systems of a wide range of equipment. These include machines for such jobs as washing, pulverizing, printing, packaging and strapping and more specialized equipment such as conveyors, welders, wind tunnels, nuclear control mechanisms, radar antennas, test equipment, and steel rolling mills.

Detailed specifications and application information may be obtained by inquiry to I-T-E Circuit Breaker Co., 1900 Hamilton St., Philadelphia 30, Pa.

Eaton

The Dynamatic Division of Eaton Manufacturing Co. in Kenosha, Wis., has just released a comprehensive illustrated bulletin describing its complete line or adjustable speed drive equipment for industrial applications, accordlng to F. L. Hopf, general manager of this electrical and electronics division.

The bulletin discusses the company's eddy-current couplings, drives, brakes, and dynamometers, as well as the magnetic friction clutches and brakes. A copy of Bulletin No. GB-3 may be obtained from Dynamatic Division, Eaton Manufacturing Co., Kenosha, Wis.

Oilgear

The Oilgear Co. and its Servocontrol Division, manufacturers of fluid power systems, have introduced a new line of electrohydraulic servo components in four bulletins just off the press.

Bulletin 36100, 8 pages, "New Electrohydraulic Servo Valves," illustrates and describes the construction and principle of operation of this plunger-less valve design.

Bulletin 36200, 4 pages, "New Power Amplifier," covers the specifications, capabilities, and operation of Servocontrol's universal type amplifier.

Bulletin 36300, 4 pages, "New Preset Units," illustrates and describes the construction and specifications of these units.

Bulletin 36400, 4 pages, "New Panels," illustrates many of the different forms and types of custom-built amplifier and control panels built by Oilgear-Servocontrol for operating electrohydraulic servo systems.

Above bulletins available from The Oilgear Co., 1572A West Pierce St., Milwaukee 4, Wis.

G.E.

GET-2929, describes in text, tables, and pictures the characteristics and application range of insulating materials for electrical insulation systems. It includes technical data on available products including mica mat, varnished cloths and papers, insulating varnishes, wire enamels, "Irrathene" irradiated polyethylene, sealing and filling compounds, insulating finishes and adhesives. The book is available from General Electric Co., Schenectady 5, N. Y.

"Helical Gear-Motors," GEA-6704, describes in text and pictures construction features and applications of helical gear-motors and the G.E. speed reducer line. Information is also provided on field and factory testing help. The booklet is available from General Electric Co., Schenectady 5, N. Y.

Shawinigan Resins

A new fine particle polyvinyl acetate homopolymer emulsion is now available in commercial quantities it has been made known by Shawinigan Resins Corp.

Designated GELVA TS-85, the new emulsion has an average particle size of 0.2–0.3 microns and an emulsion viscosity of 1400–1700 cps. at 55% solids. The emulsion is said to have excellent mechanical stability, good blocking resistance, and freeze-thaw stability and is highly stable on dilution. Tolerance to most plasticizers: borax, inorganic salts, and most organic solvents (except ethyl alcohol) is excellent, the company states.

For further information write to Dept. SN, Shawinigan Resins Corp., Springfield 1, Mass.

Houghton

A recommendation chart suitable for hanging on wall or storage bin in a maintenance shop highlights the proper application of Houghton Val-Pac rod and sheet-type compression packings.

Based on specific application conditions, this chart pinpoints the Houghton packing or packings for eight basic types of service including air and inert gases, water and neutral fluids, acids, alkalies, etc.

Write E. F. Houghton & Co., 303 West Lehigh Ave., Philadelphia 33, Pa., for a copy.

Handling 8-ft. long pulp logs is easy and economical at this Everett, Wash., mill because of a pair of pulpwood grapples that move 100,000 cords of wood per year. Approximately 720,000 bd. ft. of pulpwood is used daily at this Scott Paper Co. plant which produces more than 750 tons of quality bleached sulphite pulp and more than 300 tons of paper each day. The grapples require minimum maintenance and plant engineers say they offer the most economical way to unload pulp logs direct from railroad gondola cars to transfer decks of barking operations. One of the two Size 16 grapples, made by Blaw-Knox Co., Blawnox, Pa., is shown handling 8-ft. logs. The grapple—operating from a 7 1/2-ton, 50-ft. span bridge crane—utilizes 3/4-in. cable and is reeved with five parts of line. The grapples are equipped with style "B" equalizers to prevent twist. Each grapple has an open spread of 10 1/2 ft. Capacity of each Blax-Knox grapple is 1 cord. The closing mechanism is located well above load area to eliminate operational interference

Mt. Hope

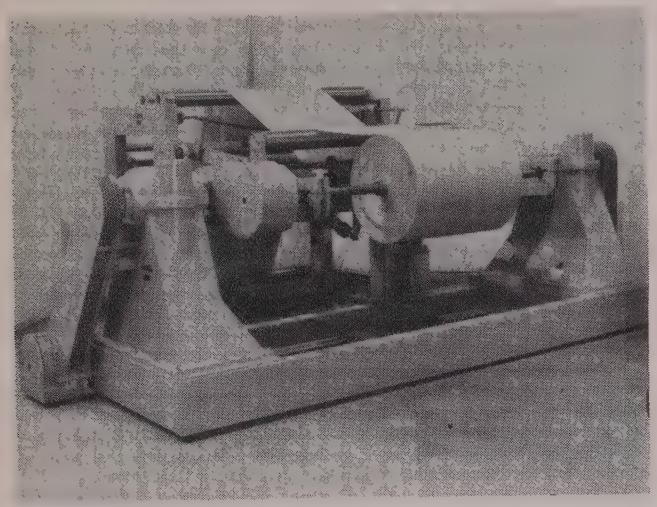
Paper mill production men, even those who now use Mount Hope rolls at the size press and in other operations, are learning new applications to calender and supercalender operations. These data are reaching them through the media of the latest in the series of technical bulletins for papermakers—"Application of Mount Hope Rolls to Calenders."

Write Mount Hope Machinery Co., 15 Fifth St., Taunton, Mass., for a copy of this new technical bulletin.

F. W. Egan

Introduction to the paper processing industry of Egan Phantom Axis unwinds and winders has been announced by Frank W. Egan & Co. of Somerville, N. J.

The machines, both of which feature a design which eliminates the center shaft, are capable of handling larger web roll diameters in about 25% less floor space and height than



Egan Phantom Axis unwind and winder

conventional turret unwinds and winders. And greater rigidity is attained from the units' compactness of design.

Now regular items in the Egan line, the new machines were standardized after an intensive, year-long development program by Egan engineers. The unwinds and winders are the only ones of their kind in regular production today.

Both machines offer speeds in excess of 1000 f.p.m. and web widths through 120 in. Other twin features are: manual or power side shifting, air chucking of cores or shafts, and knife cut-offs.

In addition, the unwinds feature a pneumatic constant tension system, flying splices at full machine speed, and a provision for splicing with either the inside or outside of the web facing upward. The winder assures smooth transfers at full machine speed and gives the operator a choice between constant and tapered tensions.

Du Pont

Results from Du Pont's engineering test center in Wilmington, Del., show that slippage of fiberboard shipping cartons, due to impact, acceleration, and deceleration, can be reduced approximately two thirds by an application of Ludox colloidal silica, a leading antiskid agent.

Seventy-five per cent of all rail damage to one-trip boxboard containers is ascribed to these causes. Railroads consider four- and five-mile-per-hour coupling impacts within the range of normal handling. Under these conditions, containers treated with Ludox proved to resist slipping most effectively.

Trials were conducted on a Conbur incline impact tester which simulates the impacts received during shipment. Single-wall, 175-lb. test corrugated fiberboard cartons were assembled and filled with 12 one-quart bottles of water, weighing 40 lbs. Cartons were sealed using a full-flap glue closure.

Use of Ludox can effect greater stability of carton loads under all shipping conditions, says Du Pont. Slippage resulting from side sway and vertical bouncing is also lowered. In addition, independent tests have established that cartons treated with Ludox can be inclined to more than twice the angle of untreated cartons before slipping begins.

Ludox colloidal silica is an opalescent, aqueous dispersion containing 30% SiO_2. Chemically, the product can be considered a colloidal solution of hydrated silica or a polymerized form of silicic acid. It can be applied without difficulty on any corrugating line.

Ingersoll-Rand

A new bulletin by Ingersoll-Rand comprehensively describes the company's pumps built for the pulp and paper industries. The bulletin, Form 7296, contains a handy table that matches each paper making service with the several models of pumps designed to handle that specific job.

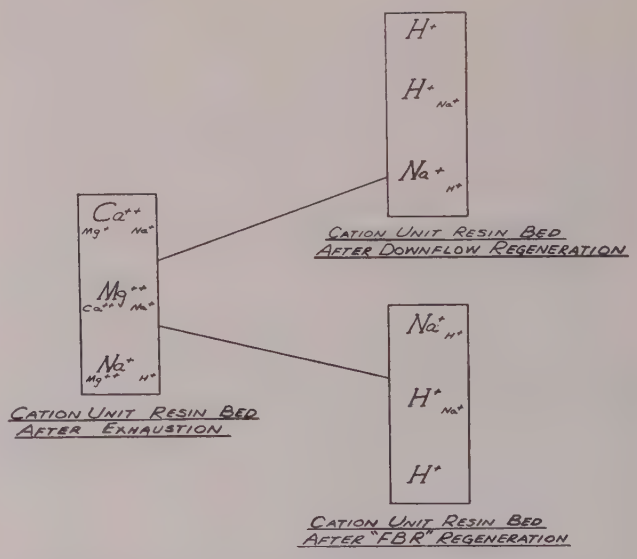
The bulletin is available from any Ingersoll-Rand Co. branch office, or from Ingersoll-Rand Co., 11 Broadway, New York 4, N. Y.

Graver

A cation regeneration method that results in lower electrolyte leakage, greater uniformity of effluent quality and reduced operating cost has been developed by Graver Water Conditioning Co., 216 West 14th St., New York 11, N. Y.

The method, known as FBR regeneration (for fixed-bed reverse flow), consists basically of a means of flowing regenerant up instead of down while maintaining a fixed, rigid resin bed. A fixed bed prevents channeling or short-circuiting of the regenerant solution.

By pumping acid up through the bed, the band of resin richest in hydrogen is at the bottom which is the point the water being treated touches last before leaving the unit. This rich band becomes a scrubber zone and reduces leakage so that a purer effluent is produced.



Contrast between conventional and "FBR" regeneration is graphically illustrated in this diagram. When cation resin bed is regenerated by downflow method, the band richest in hydrogen is located at the top of the bed. When "FBR" system is used, the richest hydrogen band is at the bottom, creating a scrubber zone for the water being treated

In the past, using conventional downward regeneration, effluent quality has been improved by one of two methods—increased acid dosage or increased bed depth. Both are continuing expenses and mean added operating costs. FBR regeneration, on the other hand, requires only one added cost—for specially engineered equipment installed in the top section of the cation unit. Operating results show that the FBR system has superior performance characteristics compared with the other two methods, says Graver.

Chief use of FBR regeneration is with high solids waters to get the purest effluent possible. However, there can be advantages in using it in connection with low solids water where it may permit a considerable reduction in equipment requirements. This application depends on various factors in the plant and only careful study will indicate its possible advantages.

American SF

Paul E. Crafton, has been appointed resident engineer in Atlanta, Ga., it has been announced by Arne Hallencreutz, president, American SF Products, Inc.

In commenting on the appointment, Mr. Hallencreutz said that "this is the latest step in our efforts to give our customers best possible service. We feel that Mr. Crafton, who has a rich experience in the air drying field, will be of much help to our customers in the South." Mr. Crafton's area of operation will include the states of North Carolina, South Carolina, Tennessee, Georgia, Florida, Alabama, and Louisiana.

American SF Products, Inc., is the United States subsidiary of AB Svenska Flaktfabriken, Swedish manufacturers of Flakt dryiers and other industrial heating and ventilating equipment.

B-C

The Flexiblade, a new patented blade-type-coating-machine for on- and off-machine clay coating, developed by The Black-Clawson Co. at its Dilts Division, Fulton, N. Y., is in operation on paper machine application.

The Flexiblade features an enclosed fountain and positive feed of coating without cascading of coating from the blade.

Coating weight can be varied and controlled by adjustments while the machine is running. It can be operated with either a positive feed or recirculation of the coating. The coating chamber is accessible for rapid blade change and clean-up, and the machine is operable over a full range of paper machine speeds.

The Flexiblade coater utilizes an inverted flexible steel blade to deliver a smooth, pattern-free coating. It handles high solids coatings which require less adhesive, result in higher brightness, and cut down drying time.

A pilot size Flexiblade coater at the Dilts Division coating laboratory, Fulton, N. Y. is available for customer demonstration and research work.

For information on the Flexiblade, write to The Black-Clawson Co., Dilts Division, Fulton, N. Y.

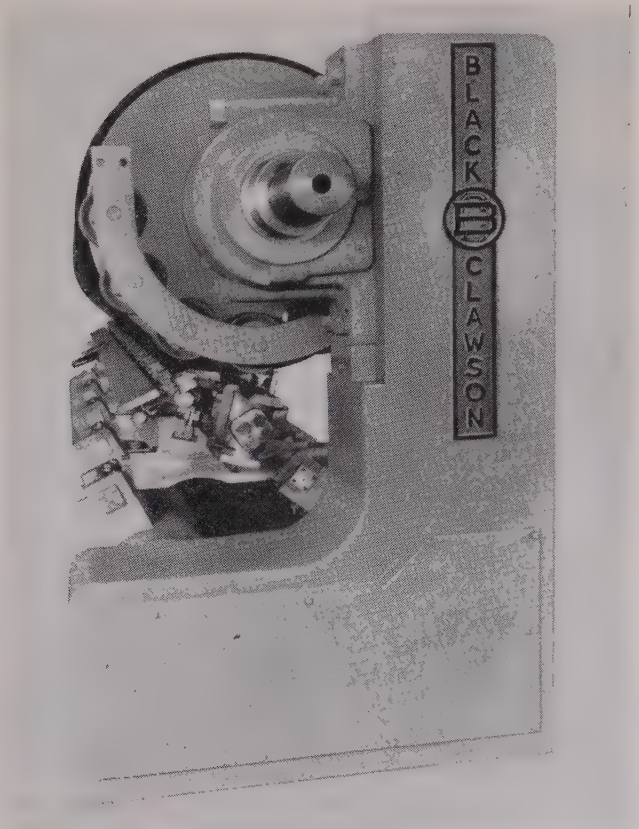
Included as an integral part of its program of new product developments for machine coating The Black-Clawson Co. has introduced an improved horizontal size press which features standardized basic unit design and balanced pneumatic loading of rolls.

Designed and built by the Paper Machine Division of Black-Clawson at Watertown, N. Y., the horizontal size press is available in roll sizes ranging from 16 in. diameter up to 36 in. diameter matched rolls.

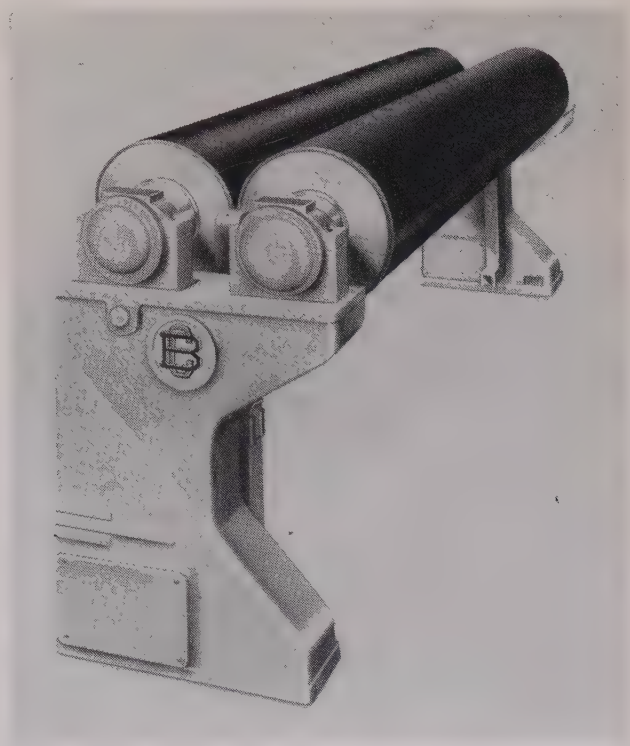
Rolls are carried in heavy duty antifriction bearings. The pivot roll arms are equipped with short-stroke pneumatic cylinders on large units and frictionless diaphragm units on smaller presses. When air pressure is released rolls separate automatically. Air cylinders are enclosed in end frames and are readily accessible through large openings under cover plates.

Modern frame styling is patterned after the proved Black-Clawson open-window drier frame design for maximum access to the traveling web, safety, and cleanliness.

The Black-Clawson horizontal size press is a standardized basic unit to which accessory equipment can be added, as required. Such accessory parts can be furnished with the



The Flexiblade coater



Black-Clawson horizontal Size press

basic unit, or can be fitted in the field to premachined mounting pads which are provided. These include: lead-on and lead-off rolls, shower pipes, hydraulic loading, carriage units which permit removal of entire unit to machine aisle for clean-up and maintenance, a selection of roll coverings and automatic features to meet individual mill requirements.

Application data and coating recommendations may be obtained from the Black-Clawson Paper Machine Division, Watertown, N. Y., or from the Dilts Division coating specialists at Fulton, N. Y.

Lamb-Grays Harbor

A precision sheet cutter-layboy engineered by Lamb-Grays Harbor Co., Inc., Hoquiam, Wash., manufacturers of sheet finishing, roll finishing, and pulp handling equipment for pulp and paper mills and converting plants, features one-man operation with all controls for both cutter and layboy centralized at the layboy.

This machine is designed for utmost cutting accuracy at speeds up to 1000 f.p.m., cutting up to 600 lb. total basis weight to a sheet-length accuracy of $^1/_{64}$ in. As a result, many trimming operations are eliminated.

Designed for minimum down time to provide maximum operating time on each shift, the machine features a multiple-roll backstand shuttle-carriage which can be shuttled from side to side. While one half of the backstands are being loaded with new paper rolls on one side, the rolls on the other side are unwinding through the cutter. This reduces time required for change-over to a few minutes only.

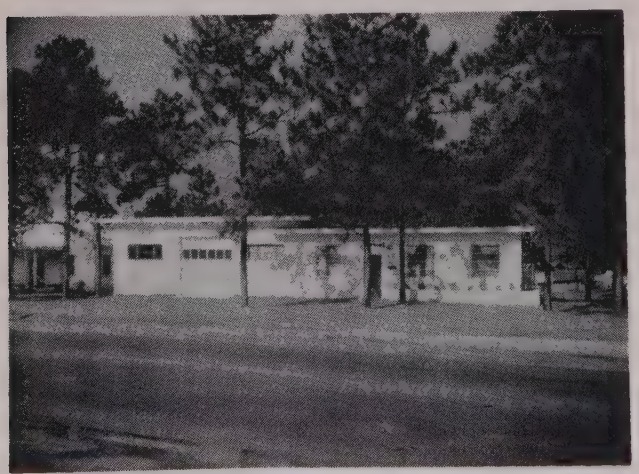
Another minimum downtime feature is the automatic layboy-discharge, which discharges a 72-in. high stack (including skid) automatically upon a receiving conveyor for lift-truck pickup. This feature avoids having to wait for pickup when a lift-truck is not immediately available.

The Lamb precision sheet cutter-layboy also features extra heavy, rugged construction for long life and minimum maintenance under continuous operating conditions. An 8-page folder fully describing the Lamb cutter-layboy is available from Lamb-Grays Harbor Co., Inc. Request Form No. 5905.

Stebbins

The increasing demands for special cements, ceramic lining material, and membrane-covered tanks and fittings, especially during emergency shutdown work in the chemical process industries and in pulp and paper mills, have dictated the need for additional facilities at the Pensacola, Fla. office of Stebbins Engineering and Manufacturing Co. Accordingly, Steve Haley, Pensacola manager, expects to complete 15,000 cu. ft. of additional membrane shop space and warehousing by May 1. A lining shop under the direction of Charles Astifan, assistant manager, will now be available for the installation of polyester, epoxy, PVC, and other nonmetallic linings in vessels of a size for truck transport.

Materials will be kept in stock to handle almost any emergency in the ceramic lining field for installation under the over-all supervision of Tom Reiter, General Southern field superintendent.



The Pensacola, Fla. office of Stebbins Engineering & Manufacturing Co.

Huber

A new mechanism, easily installed in printing cylinders of existing printer-slotter machines, will improve die registration and reduce down time, according to J. M. Huber Corp. Huber's Ink Division, exclusive sales agents, is introducing it to the shipping container industry.

Called the Dorr Rapi-Die Register, the attachment provides a quick and accurate method of fastening premounted dies without stapling or guesswork. It also makes possible the use of a new type of removable die backing.

In addition to greatly improved registration in multicolor printing, down time savings on a two-color printer-slotter of 1 hour per 8-hour shift is claimed for the mechanism. As a result, according to Huber, the cost can be amortized in about six months for the typical two-color machine.

The Dorr Rapi-Die Register is readily adaptable to the four domestic makes of printer-slotters, namely Langston, Hooper, Greenwood, and S & S. Installation can be made without interfering with the operation of the customer's printer-slotter machine. It can be made quickly in a new cylinder, or a spare one in good condition.

The Dorr Rapi-Die attachment is fastened to the metal core of the cylinder after removal of a piece of wood lagging. It consists of a square bar, called the fixed pin bar, and a circular rod, called the tightener shaft. Both the fixed pin bar and the tightener shaft have calibrated pins for accommodating grommets which are inserted in the die sheets. The

The Dorr Rapi-Die register allows for fast, accurate fastening of pre-mounted dies to printer-slotters. The mechanism eliminates the need for stapling, speeds make-ready, and assures registration of job runs and reruns

grommeted holes are accurately spaced in the sheets, parallel to the series of holes at the other side of the sheet. This enables the press operator to keep his dies on one cylinder parallel to his dies on the other. Once the dies have been properly mounted, the mounting sheets can be used over and over again on succeeding runs with assurance of register.

Development of the new mechanism was accomplished by Dorr Sales & Engineering Co. of Milwaukee, Wis., in cooperation with the Ink Division of J. M. Huber Corp. Dorr undertook this study three years ago at the request of a major box manufacturer.

This study revealed that the present practice of stapling dies to printing cylinders and the time to remove the staples after each press run or during start-ups were major down time factors. Stapling also results in inaccurate registration, laceration of cylinders, and the safety hazard of cut hands from broken staples.

The Dorr Rapi-Die Register is said to provide an engineered solution to these problems with the result that die backing and cylinder lagging can last indefinitely.

Auxiliary fixed pin bars are available, thus making it possible for dies to be used on machines of different cylinder diameters. The auxiliary fixed pin bars are installed at predetermined distances from the main unit in conformity with the sizes of sheets used in a particular box plant.

In addition to the equipment for printer-slotters, supplementary equipment for the mounting of dies in the plant die layout room is also available through Huber.

For box manufacturers with multiple plant operations, an unusual advantage of the Dorr system is the interchangeability of the die backing sheets. This permits their use not only on machines of different size and manufacture, but also from one plant to another in order to maintain an equitable balance of work loads.

Fully illustrated descriptive literature on the Dorr Rapi-Die Register can be had by writing J. M. Huber Corp., Ink Division, P. O. Box 626, Hillside, N. J.

Morningstar-Paisley

The election of Murray Stempel as president of Morningstar-Paisley, Inc. by the board of directors, has been announced by Joseph Morningstar, chairman of the board. Mr. Stempel succeeds George J. Muller, who has been elected vice-chairman of the board. Morningstar-Paisley, Inc. is a major producer of starches, gums, dextrines, adhesives, and chemical specialties for a wide number of industries including the packaging, paper, food, pharmaceutical, cosmetics, flavoring, textile, printing, mining, coatings, lithography, and tobacco fields. Consolidated sales for 1958 amounted to $23,136,122.

A new gum and technical products department, which will unify the operations of two previously distinct groups, has been formed by Morningstar-Paisley, Inc., New York, N. Y., it has been announced by Earl C. Lenz, vice-president. The consolidation of the technical products department with the water-soluble gum department will provide expanded sales and technical service to processors and manufacturers who use gums, starches, and other specialty items for stabilizing, thickening, binding, gelling, sizing, coating, flocculating, and encapsulating. According to Mr. Lenz, the new department will coordinate all technical activities dealing with gums and other specialties from the customer service standpoint at both the product development and product application levels.

Murray Stempel, Morningstar-Paisley, Inc.

T. W. Morningstar, Morningstar-Paisley, Inc.

M. P. Malkovich, Morningstar-Paisley, Inc.

Users of water-soluble gums and related specialties in a wide number of fields—food, pharmaceutical, cosmetics, flavoring, textile, paper, printing, mining, coatings, lithography, tobacco, and others—will benefit from the broader and deeper authority of the department, which includes specialists in the aforementioned industries.

Thomas W. Morningstar has been named general manager of the new department, and Mark P. Malkovich, who has been specializing in product development, has been appointed sales manager.

Morningstar's service position in the water-soluble gum field was strengthened recently by the acquisition of Thurston & Braidich, oldest gum importer and processor in the country, and the establishment of a gum processing plant in Hawthorne, N. J., which is the most modern of its kind on the continent.

Other Appointments

Miss Kay Colton has been named manager of cosmetic sales of the department, which will provide increased technical service to the cosmetic field. John Birrell will continue as manager of corrugating sales. Thurston & Braidich personnel who have been incorporated into the new department include Charles Rosevear, Jr., administrative; Charles M. Ferri, technical-field service; and George Gilbert and James Sullivan, field sales. Other key members of the department in sales assignments are John J. Quinn and Robin T. Hazell, Morningstar's Canadian sales and service representative with offices in Toronto, Ont.

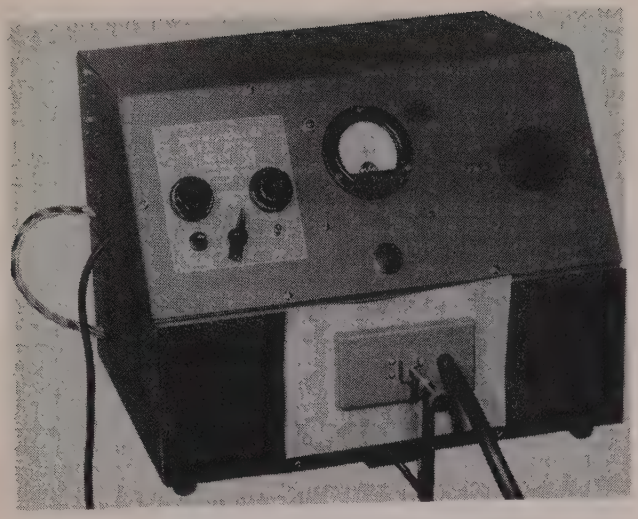
Hunterlab

A new photoelectric glossmeter has been designed by Hunter Associates Laboratory, Inc.; 5421 Brier Ridge Rd.; McLean, Va. for papers, paints, plastics, waxes, floor coverings, and textile yarns, fibers, and fabrics. Accuracy is assured by precise machine construction and assembly of all optical components. Adaptability of the instrument to both standard and new gloss tests is achieved by building elements of the incident and reflected light beams onto aluminum blocks which are mountable at either 75°, 60°, 45°, or 20°. Because of the ease with which beam and field angles can be changed, the instrument is suited for special gloss tests of new products.

The optical and electrical units are in separate housings connected by cable. The instrument can thus be used:

1. With specimen on front as shown,
2. Turned on its back with specimen mounted horizontally on top, or
3. With optical unit separate and mounted either flush in a table top for examination of large sheets or over a moving web for gloss recording.

For the paper industry, the new instrument will measure



Hunter multipurpose glossmeter

gloss of coated book papers by TAPPI 75° Method T 480 m-51, of waxed paper by TAPPI 20° Method T 653 sm-58, of low-gloss papers by contrast gloss, and of high-gloss cast-coated and varnished papers by special narrow-aperture methods. A suction-type specimen plate is available to hold paper and plastic films flat for accurate gloss measurements.

For high precision, a digital dial is used in the measurement arm of a null-balance bridge. This digital dial is operated either manually or by servo-motor.

Bauer

The Bauer Bros. Co., Springfield, Ohio, has been issued a United States patent on the use of its continuous screw press in the pulp and paper industry. This machine sold under its registered trademark "Pressafiner," is used for high pressure expression of cooking liquors or other fluids from wood chips or agricultural residues of a fiberous nature.

Ten claims were granted to the inventors, J. C. Shouvlin, president of The Bauer Bros. Co., and M. E. Ginaven, a vice-president of the company. Several claims cover the use of the machine for extraction of cooking liquors and for defibering wood chips and the like. The remainder of the claims deal with the use of screw presses as used in pulp-making processes with cookers and refiners. The full scope of the invention is described in United States pat. 2,893,909.

Appleton Machine

Hot Spot carbonized forms will be rolling off a converted Miehle vertical press during the Seventh Educational Graphic Arts Exposition in New York, N. Y., September 6–12, according to Eugene Doven, vice-president, Appleton Machine Co., Appleton, Wis. The firm's Doven Division converted the press for demonstration in its exhibit.

The conversion unit is adaptable to all standard platen and flatbed presses including the Kluge, C & P, ATF, Miller, and Miehle. Special carbonizing systems can be built for several single and multiple web rotary presses and also for offset presses.

Appleton Machine builds its Hot Spot carbonizing system into parts of the press—fountain, inker, assembly bed, and inking plate—and then returns these converted parts to the printer where they can be easily reassembled by a press machinist. Appleton offers factory help on reassembly if desired.

The conversion consists of thermal heating units with a three-point electrical control panel built into each part. Special carbonizing rolls, which withstand heat without disintegrating, are provided with the conversion.

Special inks containing a carnauba wax base have been developed for Hot Spot carbonizing. The ink melts at high temperatures and hardens at room temperature in a fraction of a second.

The carnauba wax ink has another feature of special interest to printers. It's not necessary to wash the rollers after use as long as they are not used on a different color. The wax dries and hardens making a protective coating.

A standard press converted for Hot Spot carbonizing work is still able to perform regular printing. The changeover from Hot Spot carbonizing to printing takes the same length of time it takes a printer to change from one color to another.

The Doven Hot Spot carbonizing conversion system will in no way injure or impair the performance of the press says the company.

The new Hot Spot conversion system does not give off any fumes and does not require an exhaust fan. Also the need for a spray has been eliminated. If running at high speeds, an air blast is recommended to accelerate drying.

Dow

A patented method for making liquid density gradients now can be used as an ASTM procedure for laboratory, experimental, and analytical purposes.

The Dow Chemical Co. has waived part of its rights under United States pat. 2,825,698 with the result that industrial concerns and others may practice the method without express license for the purposes specified by ASTM. Dow has not waived its rights in connection with commercial use of the method.

The patent was issued to Dow on March 4, 1958, and the inventors are William C. Taylor and Lu Ho Tung, both associated with the company's High Pressure Laboratory at its Midland Division.

The method involves transferring a liquid mixture of constantly changing density to a cylinder. Dow uses it primarily to measure the density of plastic samples. It is suitable for other materials, however.

National Starch

National Starch and Chemical Corp. has announced the purchase of Polimeros S. A., a manufacturer of vinyl acetate polymers, located in Mexico City.

Donald Pascal, president, National Starch and Chemical Corp., said that his company had been interested in the possibilities of Polimeros since its founding in 1954. National assisted in the design of the original plant and equipment and had licensed Polimeros to manufacture polymers in Mexico.

Donald E. Reese, the founder of Polimeros S. A., will continue as president of the Mexican company which will operate as a wholly-owned subsidiary.

Production from this plant has been used primarily by the paint industry for the manufacture of latex paints. Additional equipment will be installed so that a complete line of vinyl acetate polymers and copolymers, as well as adhesives for the packaging, structural, and furniture industries may be made.

National Starch and Chemical Corp. (formerly known as National Starch Products Inc.) is a leading producer of adhesives, starch products, and chemicals. Already one of the two largest producers of vinyl acetate polymers and copolymers in emulsion form in the United States, National Starch and Chemical Corp. will extend its full technical aid to the new operation in Mexico. A polymerization plant has been in operation in England since last year, and another unit at Toronto, Ont., is expected to be on stream in June.

Cleveland Crane

The Cleveland Tramrail Division of The Cleveland Crane & Engineering Co., Wickliffe, Ohio, has revised their booklet titled "Engineering and Application Data" which pertains to overhead materials handling equipment.

The booklet will be sent free upon request. Ask for Booklet No. 2008-0.

STALEY

The stockholders of the U B S Chemical Corp. at Cambridge, Mass., have voted to authorize the acquisition of U B S by the A. E. Staley Manufacturing Co., Decatur, Ill., through the exchange of $1\frac{3}{4}$ shares of U B S for each 1 share of Staley's. It is anticipated that the transaction would be completed on June 30, 1959.

The acquisition of U B S by the Staley Co. is expected to add impetus to Staley's research and polymer chemicals as well as aid in the growth and diversification of their product line. However, U B S will initially continue to operate as an autonomous unit with Paul W. Atwood continuing as its president.

RODNEY HUNT

The Rodney Hunt Machine Co. has announced the appointment of Paul Torres as sales representative for its full line of industrial rolls for paper mills.

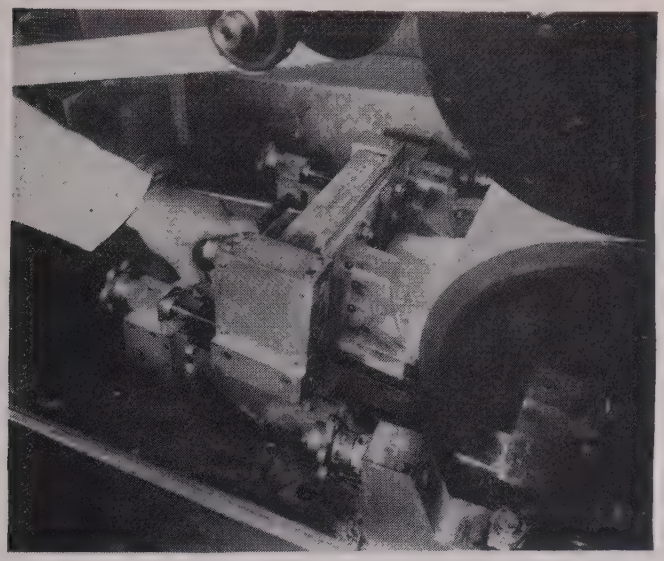
MINERALS & CHEMICALS

Minerals & Chemicals Corp. of America has recently installed a modern high-speed paper coater in their research center at Menlo Park, N. J. This equipment will be used to assist coated paper and paperboard manufacturers in the development and evaluation of present and future coating formulations and methods. This major addition makes the M&C research center one of the most completely equipped in the industry for research and development in the field of paper coating, according to C. G. Albert, director of research.

Paul Torre, Rodney Hunt Machine Co.

Top speed of the new coater is 2000 f.p.m. Built by Wheeler Roll Co. of Kalamazoo, Mich., it is approximately 20 ft. long and 8 ft. high and is located in a special room in the research center. Basically, a roll coater, the new coating machine is sufficiently flexible to duplicate most coating methods. It is equipped with a trailing blade and may be adapted to duplicate reverse roll, gravure, and air knife coating. Its infrared driers are augmented by a steam-heated Yankee drier.

This modern coater is enabling M&C research personnel to improve their study of all phases of coating technology—including coating color rheology, pigment and adhesive evaluations, performance of coating colors on the machine, calendering characteristics, and printability of coated paper. Coating basestock, supplied in 12 in. wide, 3 ft. diameter rolls, may be coated, calendered and tested for optical properties and printed on a Vandercook proof press, under conditions and with equipment closely simulating that used commercially. The coater and auxiliary equipment is expected to be especially useful in the development and evaluation of new products to meet the demands of the growing paper and board coating industries.



Close-up of the trailing blade attachment on the new laboratory coater. Angle of incidence of blade to the roll is adjustable mechanically. In addition, the angle of the entire trailing blade assembly with respect to the shaft can be adjusted through an electrical drive controlled by the knob which the operator is turning

B & W

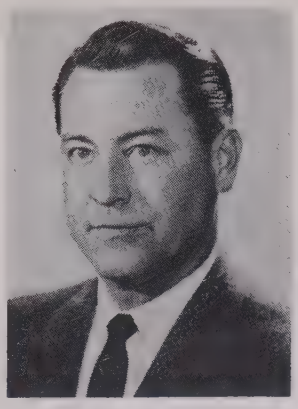
A linear conversion table for handy reference by engineers, purchasing agents, and others who have occasion to convert inches and fractions of inches into decimal parts of a foot has been issued by the Tubular Products Division of The Babcock & Wilcox Co. The table is suitable for many purposes in addition to its application to steel tubing. Known as TDC 110, the table is availbale without charge from the sales office of the company at Beaver Falls, Pa.

RAYBESTOS-MANHATTAN

To meet present-day needs for better distribution and to provide strong support to expand sales of its many specialized products, Raybestos-Manhattan, Inc., Passaic, N. J., has announced important changes in its sales structure. R. B. Parks, formerly San Francisco district manager, has been appointed assistant sales manager for industrial rubber products and packings. A new marketing group section of its Sales Division has been established. This group will coordinate all activities of marketing and work closely with production, development, sales engineering, and product sales departments.

S. J. Synnott has been appointed general marketing manager of industrial rubber products, and will direct activities of industrial rubber marketing managers assigned to specific product groups. Newly appointed members of this new rubber product group are: H. J. Seeley, marketing manager for Poly-V drives and flat transmission belts, A. Arguedas, for conveyor and elevator belts, J. J. Connors, for rubber covered rolls. This comprises the marketing group on industrial rubber products and will be expanded later to include other products. G. E. Horvath has been made marketing manager of mechanical packings.

Other changes were also made in the sales organization. R. E. Stiles replaces Mr. Park as San Francisco district manager, N. L. Rush, previously manager of distributor



R. B. Parks, Raybestos-Manhattan, Inc.

S. J. Synnott, Raybestos-Manhattan, Inc.

sales of the Philadelphia district, has been appointed Minneapolis district manager.

REICHHOLD

Plans to build a multimillion dollar melamine plant somewhere in the South have been announced by Henry H. Reichhold, president of Reichhold Chemicals, Inc. Initial capacity of the plant, which is scheduled to go on stream early in 1961, will be 20,000,000 lb. per year, but it will be designed for eventual expansion to 50,000,000 lb. annually.

"Our new plant," Mr. Reichhold stated, "will make RCI the second major supplier of melamine in the United States and we will be entering the market at a very opportune time. Demand for this chemical and for melamine molding compounds and resins is increasing very rapidly from manufacturers of plastic products, laminates, surface coatings, paper, and textiles.

"Long term research on the processes and engineering now makes possible our entry into the field and the proceeds of RCI's recent issue of 400,000 shares of new common stock have provided the funds.

"This is but one step," Mr. Reichhold continued, "in the $24,000,000 three-year expansion program upon which we embarked in 1958. There will be others."

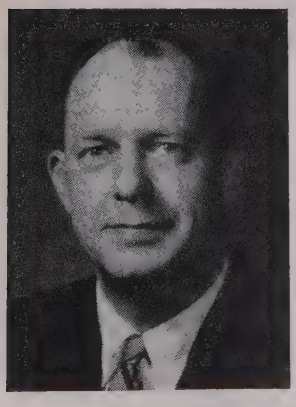
KOPPERS

Harry B. Cummings, vice-president of Koppers Co., Inc. and general manager of its Metal Products Division, Baltimore, Md., announces that the division's F. X. Hooper Box Machinery Department, Glenarm, Md., and George W. Swift, Inc., Bordentown, N. J., have been combined to form the Container Machinery Department of the division. The announcement followed disclosure last week of the purchase by Koppers of all assets of the Swift Co., producers of machinery for the corrugated container industry.

Mr. Cummings also announced the promotion of Charles D. Nitchie, formerly chief engineer at Hooper, to the position of general manager of the department which will operate as a product line organization. Mr. Nitchie will have complete responsibility for marketing, finance, production, and engineering for the department. He will be responsible to Mr. Cummings for the profitable management of the container machinery business and for the operation of the department within the framework of division policy and procedures.

E. D. JONES

Donald W. Curtis has been transferred from Beloit Iron Works, Beloit, Wis., to the E. D. Jones Corp., Pittsfield, Mass., as assistant to the president. In his new position he is taking active part with other members of the Jones executive staff in plans to expand the company's facilities and service to the papermaking industry.



D. W. Curtis, E. D. Jones Corp.

J. F. Voit, Ralston Purina Co.

RALSTON PURINA

Joseph F. Voit has been named sales manager of the Special Soy Products Division for the Ralston Purina Co. and has assumed his duties at the company's general headquarters in St. Louis, according to an announcement by Vice-President Eldred A. Cayce.

W. E. Tjossem is manager of the recently created Special Soy Products Division. This division produces and supplies isolated soya protein to the paper industry under the trade name Purina Procote.

OBITUARIES

John King Ferguson

John K. Ferguson, resident manager of the St. Regis Paper Co. mill at Jacksonville, Fla., died in Pensacola, Fla., of complications following surgery.

Mr. Ferguson was born at Great Neck, N. Y., on March 6, 1924, and attended Syracuse University. He was the son of Roy K. Ferguson, chairman of the board of the St. Regis Paper Co.

Erik Hagglund

Professor Erik Hagglund died in Stockholm, Sweden, on March 13, 1959. Dr. Hagglund was professor of pulp, paper, and wood technology at the Royal Technical University of Sweden. He was also the head of the Wood Chemistry Department of the Swedish Forest Products Research Laboratory.

Professor Hagglund was born in Hassjo, Sweden, on June 15, 1887, and received his doctorate from the University of Stockholm in 1914.

From 1911 to 1915 he was chemist for the Bergvik Sulphite Pulp Co. in Bergvik, Sweden. From 1916 to 1920 he was a department manager for Th. Goldschmidt A/G. in Essen, Germany. From 1920 to 1930 he was professor of chemistry and chemical technology of forest products at the Academy of Abo in Finland and since 1930 he was a professor at the Royal Technical University in Stockholm.

Dr. Hagglund was the author of more than 100 technical papers and participated in some of the fundamental research conferences held in the U.S.A. and Canada. He became a member of the Technical Association of the Pulp and Paper Industry in 1938.

George Lawrence Witham

George L. Witham, Salesman for the Orr Felt & Blanket Co., Mobile, Ala., died from a heart attack at Springhill, Ala., on May 19, 1959.

Mr. Witham was born in Millinocket, Me., on Jan. 31, 1903, and attended the University of Maine. He was employed for more than 12 years by the Bogalusa Paper Co., Bogalusa, La., as a paper machine operator and for one year by W. C. Hamilton & Sons, Miquon, Pa.

In 1938 he became a salesman for Ciba Co. at Charlotte, N. C., and in 1945 became the south central U.S.A. representative for the Orr Felt & Blanket Co. at Mobile.

Mr. Witham became a member of the Technical Association of the Pulp and Paper Industry in 1928.

TAPPI DIVISIONS AND COMMITTEES

Reports of Activities

Corrosion Committee

In preparation for the forthcoming 14th Engineering Conference to be held Oct. 12–15, 1959, at the Penn Sheraton Hotel in Pittsburgh, Pa., E. W. Hopper, chairman of the Corrosion Committee, called an interim meeting of the committee June 2, 1959, at the Pittsburgh Athletic Association.

The Corrosion Committee is planning a panel discussion "Overlay of Alkaline Pulping Digesters" as one of two sessions to be sponsored by this committee and this was the subject of the interim meeting.

The following members and guests were in attendance:

Members

E. W. Hopper, Chairman, Consultant, Pittsburgh, Pa.
H. W. Fritts, Secretary, ALCOA, New Kensington, Pa.
Z. S. Blanchard, The Portland Co., Portland, Me.
H. M. Canavan, Mutual Boiler & Mach., Waltham, Mass.
E. G. Holmberg, International Nickel Co., New York, N. Y.
A. R. Meyer, Graver Tank, Chicago, Ill.
J. A. Morrison, Crucible Steel Co., Waltham, Mass.
W. B. Parker, Hartford St. Boiler, Hartford, Conn.
F. D. Whitehead, Jr., West Va. P. & P. Co., Covington, Va.
P. Zouck, Alloy Cladding Co., Baltimore, Md.

Guests

W. C. Barnwell, (Gaylord Div., Crown-Zellerbach), Bogalusa, La.
M. D. Bellware, International Nickel Co., New York, N. Y.
C. M. Carlisle, Jessop Steel Co., Washington, Pa.
W. G. Gerstacker, Nat'l. Annealing Box Co., Washington, Pa
F. W. Hank, West V. P. & P. Co., Charleston, S. C.
D. Innes, International Paper Co., Mobile, Ala.
G. E. Linnert, ARMCO Steel Corp., Baltimore, Md.
G. F. Lockeman, Procter & Gamble, Cincinnati, Ohio
G. R. Pease, International Nickel Co., Bayonne, N. J.
S. I. Reames, Hudson Pulp & Paper, Palatka, Fla.
R. S. Rote, Lukens Steel Co., Coatesville, Pa.
E. K. Scholz, West Va. P. & P. Co., Covington, Va.
D. T. Smith, Jessop Steel Co., Washington, Pa.
C. V. Spangler, J. F. Pritchard & Co., Pittsburgh, Pa.
R. C. Stamm, W. Va. P. & P. Co., New York, N. Y.
H. O. Teeple, TAPPI, New York, N. Y.
R. Timko, Jessop Steel Co., Washington, Pa.
J. Viera, Royal-Globe Ins. Co., Monroe, La.
A. E. Williamson, Chicago Bridge & Iron, Pittsburgh, Pa.
S. S. Wyndham, Royal-Globe Ins. Co., New York, N. Y.

The agenda for the discussion consisted of the following items:

I. Introduction of Corrosion Committee Members and guests.
II. Purpose of meeting
  (1) To set up panel discussion on overlay for 14th Engineering Conference, Oct. 14, 1959, 2:00 p.m.–5:00 p.m.
    Panel: Z. Blanchard, Portland Co., Portland, Me.; H. M. Canavan, Mutual Boiler & Machinery Ins. Co., Waltham, Mass.; E. G. Holmberg, International Nickel Co., Inc., New York, N. Y.; D. Linnes, International Paper Co., Southern Kraft Division, Mobile, Ala.; W. H. Parker, Hartford Steam Boiler & Ins. Co., Hartford, Conn.; P. Zouck, Alloy Cladding Co., Baltimore, Md.
  (2) Discuss all phases of overlay—good and bad.
  (3) Set up field and laboratory investigations to determine causes of overlay failure.
  (4) Set up procedures to follow in overlay installations.
III. Suggested topics for consideration.
  (1) Methods of overlay application.
    a. Manual arc.
    b. Gas shielded.
    c. Flux shielded.
  (2) Chemistry and metallurgy of overlay 308, 310, 316, Inconel, 330.
  (3) Chemistry of cooking liquors.
  (4) Locations of failures in digesters.
  (5) Previous attacks on welds-in carbon steel and clad digesters.
  (6) Effects of weld cracks.
  (7) Effects of slag inclusions.
  (8) Effects of gas porosity.
  (9) Effects of dilution of weld metal with carbon steel.
  (10) Effects of penetration of weld metal.
  (11) Effects of current density.
  (12) Effects of speed of overlay.
  (13) Effects of temperature build-up on overlay and base metal.
  (14) Effects of gas purity on weld metal.
  (15) Effects of insufficient gas shielding.
  (16) Effects of ferrite in austenitic overlay.
  (17) Effects of carbides on austenitic overlay.
  (18) Suggestions for laboratory investigation.
  (19) Suggestions for field investigation.
  (20) Recommendations for overlay procedures at this time.

After the all-day discussion, the many comments may be summarized as follows:

(1) Manual arc overlay properly applied has given excellent service, some still in good condition after 7 to 9 years. Some have also failed, generally where thin and heavily diluted.

Corrosion Committee Meeting, Pittsburgh, Pa., June 2, 1959

Gas shielded has had some good service but a number of failures. Many coupons examined have had microfissures.

Flux shielded has had less service experience. Some coupons tested have shown microfissure.

(2) Chemistry of overlay does show marked dilution. Therefore, for the type of corrosion in a digester we must start with an alloy sufficiently high to allow for dilution. Sulphur and phosphorous are dangerous and should be absent from the weld deposit.

The metallurgy of overlay showed nonuniformity in the cast structure of the weld metal with some areas running high in carbon steel stringers and dilution. Different areas etch differently indicating differences in probable corrosion resistance.

(3) Chemistry of cooking liquors varies from mill to mill and in many cases within mills. A test program on prepared coupons, therefore, should consider field tests in digesters at several mills.

(4) Overlay is installed on domes, cones, and side walls of digesters. Overlay just as carbon steel in some cases has shown attack on domes, cones, and side walls depending on the mill involved.

(5) Attack on the welds in clad digesters seemed to develop in welds where microfissures were present. This condition, in some mills, was corrected by developing a weld metal free from microfissuring. This was based on the use of an analysis with up to 11% ferrite content.

(6) The concensus of opinion of the group indicated that microfissuring is a major cause for weld overlay failure.

(7) Slag inclusions were not considered as causing overlay failure or microfissuring.

(8) Gas porosity was considered detrimental and possibly aggravated microfissuring in the overlays installed using gas shielding. Oxygen was considered bad due to oxide formation causing dirty weld metal, nitrogen because it is a strong austenite and nitride former, and hydrogen, especially bad, due to stresses it introduces.

(9) Dilution of the overlay with carbon steel was considered a very serious contributor to overlay attack and failure. Tests run on manual arc overlay which had good service life showed about 15% dilution, tests run at the same time on manual arc overlay coupons with 30% dilution showed quite rapid attack. However, the 30% dilution coupons were very thin, actually about $^1/_2$ the thickness of the 15% dilution coupons. Examinations of other manual arc coupons have shown from 25 to 28% carbon steel dilution in stainless type 310 with the test coupons showing no microfissuring and only slow general attack and thinning. Some automatic welded coupons have shown 50 to 58% dilution and severe microfissuring. Some of the fissures were large enough to see with the naked eye on polished sections. I have not examined any automatic overlay coupons removed after service in pulping digesters. I do have several automatic coupons on hand now that have been exposed.

(10) Penetration of the weld metal into the base metal was considered a source of dilution and, therefore, deep penetration was considered detrimental.

(11) Current density in weld overlaying was discussed. Too high current density was considered likely to cause deep penetration into the carbon steel and, therefore, high dilution. Current densities, however, would change with the different methods of welding and sizes of rod and wire used.

(12) Speed of overlay was also discussed. It was stated in the meeting that some of the early automatic overlay was done at speeds ranging up to 120 f.p.m. and that this speed was too high. It was also stated that speeds should be kept down to about 50 f.p.m.

(13) There was a difference of opinion on the effect of temperature build up in the base metal and the weld metal. Most of the persons who commented felt that it was detrimental, caused microfissuring, and dilution. Several persons

Corrosion Committee panel—seated: W. B. Parker, E. W. Hopper, H. W. Fritts, E. G. Holmberg. Standing: Z. Blanchard, H. M. Canavan, D. Inness, P. Zouck

took the opposite view and stated that temperature build up acted as a stress reliever and reduced cracking. There was a lack of agreement on this point.

(14) Gas purity was considered a factor in gas shielded overlay welding. While pure argon gas was stated to have poor operating characteristics, it was considered better than using argon plus 2% oxygen. It was also felt that nitrogen should be avoided as it pushed the alloy to the austenite side. One person stated, "Why use oxygen or nitrogen in the gas shield when you are trying to keep air away?" The addition of some helium to the argon was discussed briefly with no definite recommendations.

Moisture was also considered detrimental. Therefore, walls should be dry during welding. Water cooled heads should be avoided due to water leakage.

(15) Insufficient gas shielding was considered detrimental to good weld overlay practice.

(16) The presence of ferrite in the weld metal was considered seriously. It was stated that the alloy compositions discussed, namely, 13 to 19% chromium with 9 to 15% nickel should be entirely austenitic. It was also pointed out that the weld metal is not uniform throughout and, therefore, these alloy concentrations may not hold throughout. Therefore, ferrite or martensite might be present. It was stated that there was no evidence to show that ferrites were contributory to attack. This should be checked.

Photomicrographs of weld overlay sections were shown at this point. These pointed out the nonuniform weld structure, the presence of iron stringers and highly diluted areas, the microfissures, and to the difference in etching patterns both with oxalic acid and kraft liquor electrolytic etches.

Other photomicrographs showed a pattern of surface attack on coupons removed from digesters. Some of this attack appeared to be concentrated on a grain boundary phase. This will be checked further.

(17) No concrete evidence was presented to show that precipitated carbides were contributory to weld overlay failure. However, it was reported that a certain laboratory investigation had shown that a very grainy type of weld overlay failure was partially due to intergranular attack on precipitated carbides. It is hoped that this will be clarified.

(18) Laboratory investigations will be carried on to try to develop the basic reasons for attack on overlay. Several companies have promised help in these studies. Certain of the paper companies have furnished trepanned coupons or are preparing to make them available. These will include coupons from overlay with long, good service life, and some suffering from rapid attack.

(19) Field investigations may also be initiated. Test coupons for installation in digesters may be prepared using different alloys and welding procedure. These would then be available for inspection and examination.

(20) The entire subject was reviewed with the idea of developing recommended procedures to follow in installing overlay by the different processes. It was definitely suggested that there should be a minimum weld metal thickness and that this should be measured across the thinnest overlay points, namely, top of the carbon steel peaks extending into the overlay and the bottom of the valley between the beads. This should also be defined in terms of pounds of metal deposited per square foot.

(21) While preparation of the digester surface was not considered important in the earliest applications of type 310 stainless overlay, greater care and attention are certainly required with the present trend toward the use of automatic welding. The sheels surface should be clean and free from scale, pulp, oil and grease, and pulping chemicals. This cleaning can probably be best performed by sand or shot blasting. The walls should be dry before welding is started. Sharp and deep pits, grooves, welds, etc. should be smoothed and rounded by grinding or chipping.

(22) It was also stated by several of the mill personnel, that two weld bead overlays gave good service life.

Further, it was decided that maximum weld speed, current density and dilution should be specified. The only one, however, which was spelled out was a maximum travel speed of 50 f.p.m.

Beads should be close together so that base metal is not exposed. The beads should overlap so that the minimum thickness across the overlay is $^1/_8$ in. However, a real smooth overlay surface was not considered necessary for good weld life.

In conclusion, it is hoped that this meeting will stimulate thought and study on the part of the mill people, the installers of overlay, the metal suppliers, and the technical representatives on our Corrosion Committee. Overlay has worked too well in too many mills to allow the failures which have occurred to detract from its value to the alkaline pulping industry. Until new specific procedures for installing overlay can be developed, any overlay installed should be carefully installed recognizing the conditions and effects outlined above.

After overlay, the surface should be inspected. Cracks, craters, slag, and other defects should be removed. These areas and other thin or unwelded spots should be rewelded.

In conclusion, the following arrangements were made for the panel discussion at the conference:

The six panelists will be allowed one hour for the presentation of their prepared material on the subject. This will allow each panelist about 8 to 10 minutes time and you should be prepared to cover your topic in that time. I would like to have copies of your prepared discussion here by August 1 so that it can be reviewed with respect to the other topics.

The topics for discussion by the respective panelists would be as follows:

1. H. M. Canavan—Present the up-to-date picture of alkaline pulping digester corrosion based on the latest reports with emphasis on the trend in severity, location in the vessels, and the effect of the different procedures for overcoming attack on the corrosion rates.

2. W. B. Parker—Discuss the history of overlay, the preparation and procedures used in the early installations, the results of the inspection of those overlays, and the change over from manual arc to automatic overlay.

3. D. Innes—Present the attitude of the paper and pulp industry on overlay, its use, and the economics involved.

4. Z. Blanchard—Talk on the installations of overlay he has inspected both the satisfactory installations as well as the failures, the types of failures noted, the laboratory and field investigations his company has carried on, and his suggestions on procedures to follow in installing overlay.

5. P. Zouck—Discuss the field installations he has followed, the results of the laboratory and field investigations, and his suggestions on steps to follow to secure the best service life of overlay.

6. G. Holmberg—Discuss the International Nickel Co.'s laboratory investigations on overlay, the results with various alloy compositions, the possible mechanism of attack in the alkaline digester service, and the interpretation of the laboratory results.

Following the prepared presentations there will be approximately an hour for questions and answers, inspection of test coupons, and presentation of pictures and slides.

It is expected that the final hour will be devoted to concrete recommendations on procedures to follow in preparing the surfaces of the digesters for overlay, applying the overlay, repairing overlay installations, finishing up and inspecting the overlay, and to the proper chemistry and metallurgy of the metal to be used.

Fluid Mechanics Committee

The Fluid Mechanics Committee, Engineering Division, met in Parlor E of the Hotel Commodore, New York on Feb. 24, 1959, at 12:30 p.m. for a luncheon meeting, with Mr. Shoumatoff, chairman, presiding.

The following members were present:

N. A. Ednie, E. I. du Pont de Nemours & Co.
H. T. Fisher, Ontario Paper Co., Ltd.
W. L. Ingmanson, The Institute of Paper Chemistry
C. A. Lee, Newtown, Conn.
J. D. Lyall, Armstrong Cork Co.
K. J. Mackenzie, Eastman Kodak Co.
J. Mardon, Anglo Paper Products, Inc.
S. G. Mason, Pulp and Paper Research Institute of Canada
J. D. Parker, Beloit Iron Works
N. M. Reitzel, Rice Barton Corp.
N. Shoumatoff, West Virginia Pulp & Paper Co.
J. A. Smith, Clark & Vicario Corp.
P. E. Wrist, The Mead Corp.

Present also were the following guests:

D. W. Appel, University of Kansas
J. L. Ericksen, Johns Hopkins University
C. S. Yih, University of Michigan
G. Bugliarello, Massachusetts Institute of Technology

At the chairman's suggestion the minutes of the previous meeting in Portland, Ore., were accepted unanimously without reading. They had previously been accepted for publication in *Tappi*.

Fluid Mechanics Committee

Also minutes of meeting of the Subcommittee on Free Surface Flows at the University of Michigan were not read, but the chairman of the subcommittee, Mr. Lee, made a brief report covering this meeting for the benefit of those members who were not present. Excellent progress was reported by Mr. Lee of the work being done under this project under the supervision of Mr. Yih. Further details regarding this meeting will be found in the minutes by Mr. Lee.

The next subject discussed included the topics for presentation at the next meeting of the Engineering Division in October, 1959, at Pittsburgh.

Mr. Fisher reported that at this time it was not certain whether his paper on "Friction Loss Due to Excessive Smoothness" would be available in time for this meeting. Results to date, he indicated, point to the friction loss due to this factor as being more normal than had previously been believed. He expects results should be known in the near future.

The Pittsburgh program will also include a complete report covering this year's progress at M.I.T. under TAPPI Research Project No. 147 on the effects of fibers on velocity distribution, turbulence, and flow resistance of dilute suspensions. This report will be presented by J. W. Daily and G. Bugliarello of the Massachusetts Institute of Technology. Mr. Ingmanson suggested a possible paper by Mr. Han of Beloit Iron Works in the field of forming machines. The chairman mentioned the possibility of a joint session of the Fluid Mechanics Committee and the Mechanical Engineering Committee at the Engineering Division meeting at Pittsburgh. This possibility will be further explored. Mr. Lee reported a conversation with Mr. Davis of Kimberly-Clark which indicated it might be too early to finalize on a joint session between the Fluid Mechanics and Mechanical Engineering Committee, at the forthcoming meeting at Pittsburgh.

Mr. Shoumatoff announced that C. S. Yih had recently been awarded a National Science Foundation scholarship to Cambridge University for next year where he will have the opportunity of pursuing further investigations in this field in association with Sir Geoffrey Taylor. A progress report of TAPPI Research Project No. 156 will be submitted by Mr. Yih and Mr. Spengos at the Pittsburgh meeting.

The discussion then turned to subcommittee activities. Mr. Mackenzie stated that the membership of the Pipe Friction Subcommittee would be worked out by the Fall meeting.

Mr. Lee, in his capacity as chairman of the Data Sheets Subcommittee, suggested that subcommittees supervising projects at University of Michigan and Massachusetts Institute of Technology should examine the possibility of developing the results in data sheet form. It was also suggested that the previously published papers by Mr. Ingmanson and Mr. Mason in the fields of "Flow through Porous Media and Properties of Fiber Suspensions" might also be suitable subjects for data sheets. Mr. Mason suggested that it might be premature to finalize data for Massachusetts Institute of Technology project in data sheet form at this time. Mr. Shoumatoff suggested the subject of the velocity probe as developed under the Massachusetts Institute of Technology project as a suitable subject for a data sheet.

Mr. Mardon suggested that data sheets might be worked out as appendices for the final reports under these projects.

Mr. Lee distributed data sheets prepared by the Hydraulics Committee to the committee members.

Mr. Mason reported on the progress of the work at the Massachusetts Institute of Technology and stated that results to date have confirmed data obtained from other investigators. The real value of this work, he pointed out, is just about to emerge. A subcommittee meeting has been arranged at the Massachusetts Institute of Technology early in April. Mr. Ingmanson seconded Mr. Mason's motion that the subcommittee for such projects be kept at a small and workable level. Further work will be done in the field of correlating velocity and turbulence requirements. The work of Mr. Bugliarello in thesis form will be available in a couple of weeks.

Mr. Wrist mentioned that the question of submerged nozzle discharge under water had been discussed originally as part of this project. Mr. Bugliarello stated that this subject would be taken up, if time permits. He also reported that Mr. Troutman's master's thesis will be available in printed form in a couple of weeks.

Mr. Lee then reported on the progress of the Subcommittee on Free Surface Flows project at the University of Michigan. He stated that the existing flume will be rebuilt and that experiments with water would continue with work involving fibers at a later date. Mr. Yih stated that the new flume would give cleaner disturbances. Mr. Yih and Mr. Mardon discussed the subject of wave formations and also the study of motion pictures depicting wave formation and disturbances. It was concluded that direct experimental observation is probably more satisfactory for the purposes of Mr. Yih's analysis.

The report of the meeting of the Subcommittee on Free Surface Flows at the University of Michigan on January 9 prepared by Mr. Lee was accepted by the committee.

Next the discussion concerned the monograph on fluid mechanics as previously outlined during the committee meeting at Portland, Ore., in July. Mr. Shoumatoff reviewed the origin of this monograph in the remarks by Mr. Curtis last year as to the possible scope of the Fluid Mechanics Committee. The subject was then thrown open to the membership for comment. Various members questioned the advisability of diffusing committee scope into such fields as pulping or converting which, it was pointed out, would require the close cooperation and possibly review by other committees.

It was, however, also pointed out that one of the purposes of the monograph was in a sense to define the scope of the committee. As an example, the survey of the status of fluid mechanics in the paper industry prepared by Professors Ippen and Daily of the Massachusetts Institute of Technology was cited. Various time limits for completing this monograph were outlined and periods of 1 year or 6 months were mentioned. Mr. Reitzel referred to a letter to Mr. Shoumatoff in which he had outlined the possibility of preparing a monograph in terms of process differentiation rather than in terms of fluid mechanics problems.

The form and shape of the monograph as well as the scope and period required to complete are matters in which all members of the committee are interested and, therefore, Mr. Lee suggested that the committee officers draw up an outline of a possible new scope of such a monograph and that this outline be resubmitted to the committee members for approval. Mr. Ingmanson moved to adopt Mr. Lee's suggestion and the committee voted to do so.

The meeting was adjourned at 1:55 p.m.

Industrial Engineering and Materials Handling Committee

An interim meeting of the Industrial Engineering and Materials Handling Committee and its Data Sheet Subcommittee was held on May 18, 1959, at the Sheraton Hotel, Philadelphia, Pa. Those present were Vice-Chairman D. D. Hall, Secretary H. A. Stoess and members C. E. Green, J. M. MacBrayne, C. Reagan, H. F. Parker, L. Pearson, J. F. Sweers, and F. G. Wilson. H. O. Teeple of TAPPI Headquarters was also present.

In the absence of Chairman Kendall who sent his regrets at not being able to attend, Mr. Hall acted as chairman for the Materials Handling Committee meeting and also chaired the meeting of the Data Sheet Subcommittee.

Mr. Hall first discussed committee Date Sheets, and, requested information as to their status. Mr. Teeple is to supply the requested information. These data sheets were entitled as follows:

A. Pulpwood characteristics (number of sticks per cord).
B. Pulp storage capacities.
C. Industrial truck specifications.
D. Standard sizes of noncaptive pallets.
E. Determination of salt cake storage facilities—discussion followed this in that possibly this data sheet should be part of a general data sheet on bulk storage of chemicals.
F. Bulk process flow symbols—submitted to review board.
G. Flume capacities—submitted by Aux Schnyder, approved by the Industrial Engineering & Materials Handling Committee. Creed Reagan was given the job to arrange this data sheet in proper form.
H. Amortization table, prepared by Dan Patch—it was decided to refer this to the Economics Committee.

Mr. Hall then discussed data sheets now in progress, and, these data sheets are as follows:

A. Units of measure by Messrs. Reagan and MacBrayne. Discussion on this data sheet centered on its application to our committee, and whether or not a general data sheet or a data sheet for specific material would be required. Mr. Hall further suggested that possibly a questionnaire to the mills might be the answer on this data sheet. Mr. Teeple advised that it was time-consuming to handle a questionnaire, since TAPPI Headquarters has no specific mailing list prepared for such a group of mills. This data sheet should be set up as a project, and, submitted to the chairman of the committee who in turn should submit same to the chairman of the Engineering Division. The questionnaire is to be drawn up by Mr. Reagan, and submitted to members for comments.

B. Densities of bales of wastepaper. Mr. Reagan described this as a data sheet to list volumes occupied by broke in bales. It was further suggested to get additional information from the board, deinking and corrugating committees. Mr. Wilson then suggested that wastepaper dealers be contacted, since they probably would have rather good information. Mr. Pearson was designated to cooperate with Mr. Reagan on fine papers, Mr. Lawler of Union Bag would work on corrugating, possibly W. D. Boggess on deinking. Mr. Reagan is to prepare the draft of a letter to be sent to other TAPPI committees re this problem.

C. Pulpwood characteristics is to be taken over by Mr. Wilson, and, is to include the cubic feet of solid wood per cord and other characteristics of pulpwood.

D. Pulp volumes versus densities by F. D. Helversen, it was decided that Mr. Kendall should take over this data sheet from Mr. Helversen.

E. Unit weight per cord of chips by H. A. Stoess who advised that considerable correspondence was had with APA, and, that nothing definite was arrived at to date, since figures varied all over the lot. Further discussion was had on this and it was thought that possibly a questionnaire to various mills may give us more definite information.

This committee's program for the Pittsburgh meeting was then discussed, and, it was announced that we had one paper by a man from U. S. Steel Corp. in Pittsburgh and, we were having difficulty in getting the second paper. Broke handling was discussed further, and, Messrs. Wilson and Sweers were to talk with a man from Glatfelter's on the broke handling system employed by this mill. [Note: Since the committee meeting, Mr. Stoess talked with Mr. Sweers on the phone, who advised that this paper will be presented by R. D. Owen, project engineer, P. H. Glatfelter Co., Spring Grove, Pa. with a tentative title of "Broke Handling at Glatfelter's."]

Mr. Teeple announced that the session for this committee would be on Wednesday October 14, from 9:00 a.m. to 12:00 noon in the Urban Room at the Penn-Sheraton Hotel in Pittsburgh, with the committee luncheon meeting scheduled for Monday 12:30 p.m. in the Park View Room of the same hotel. It was announced that the 1960 meeting would be held in Jacksonville, the 1961 meeting in Washington, and the 1962 meeting will be held in Montreal.

A data sheet on the characteristics of materials, taken from one of the trade publications was discussed, and, it was decided to narrow this table down to materials used in the pulp and paper industry, and, this job was given to H. A. Stoess to perform.

Messrs. Hall and Reagan then discussed new data sheets, as follows:

A. Applications in pulp and paper industry for various standard material handling equipment—L. Pearson.
B. Unit man hours required for various common material handling operations in pulp and paper mills—to be tabled.
C. Fork truck travel and handling times—no action.
D. Roll change frequencies for various roll types and diameters—to be tabled.
E. Lineal feet of paper per roll—Mr. Reagan.
F. Roll weights per inch of trim for various diameters—Mr. Sweers.
G. Pulpwood storage factors—cubic feet per cord for various methods of storage—Mr. Green.
H. Freight car loading patterns and capacities for board and paper rolls of various sizes—to be tabled.
I. Truck loading patterns and capacities for board and paper rolls of various sizes—to be tabled for the present, with the possibility of a future paper.
J. Winder sets per reel for various diameters—Messrs. Reagan and Sweers.
K. Storage and shipping aisle width requirements for various size and type trucks and loads—B. C. Kendall.
L. Chart and diagram covering normal work place area (normal and maximum reach for right and left hand, proper work table height, chair height, etc.)—A. T. Tseng, Diamond-Gardner, Middletown, Ohio.
M. Tractive effort on inclines and ramps—Mr. Reagan.
N. Factors of plant layout—P. Schnyder. Mr. Teeple suggested that since this subject covered so many items that possibly a monograph should be considered. He recommended that Chairman Kendall consider the suggestion and obtain an expression of the whole committee.
O. Standard performance for fork lift trucks was suggested by Mr. Sweers. He was asked to report to the committee in Pittsburgh on whether this should be a data sheet or a monograph.
P. Attempt to set up by MTM (Methods Time Measurement) standards, suggested by Mr. Sweers—tabled.
Q. Standard industry wide allowances for fatigue, personal delay, unavoidable delay, and other factors, suggested by Mr. Sweers—tabled.
R. Unloading of pulpwood from cars and trucks (time allowances) suggested by Mr. Sweers—tabled.

The meeting adjourned at 4:10 p.m.

H. A. Stoess, Jr., *Secretary*

Alkaline Pulping Committee

The meeting was called to order at 2:15 p.m., Sunday, Feb. 22, 1959, in Parlor E of the Commodore Hotel by Chairman J. Mc. K. Limerick, with the following members and guests present:

R. H. Stevens
D. C. Gillespie
D. O. Adams
W. B. Stengle
H. O. Teeple, *Guest*
V. Woodside
T. C. Bannister, Jr.
D. J. MacLaurin
P. H. West
M. B. Pineo
K D. Running
D. Wetherhorn
J. O. Parrott
R. R. Fuller
E. Rice, *Guest*
J. Mc K. Limerick
P. B. Borlew
H. Y. Charbonnier
A. W. Pesch

On motion by Mr. Charbonnier, seconded by Mr. Wetherhorn, the minutes of the previous meeting were accepted.

Resignation of M. N. May was accepted and D. J. MacLaurin appointed in his place. Mr. MacLaurin was welcomed to the committee by the chairman.

Mr. T. T. Collins resigned due to company commitments. On motion by Mr. Wetherhorn, seconded by Mr. Parrott, the resignation was accepted.

Alkaline Pulping Committee

Chairman Limerick discussed the possibility of a representative from Dryden Paper Co. W. E. Haviland was suggested as a committee member. It was decided he would be approved.

The possibility of asking the Pulp Purification Committee to add a day to the Jacksonville program was discussed. H. O. Teeple reported that Ferd Kraft had four possible papers lined up on Pulp Purification which might be added to the Jacksonville meeting. The Semichemical committee will participate in the Jacksonville session. Mr. McLaurin as Secretary of Pulp Purification considered doing some contacting of the Dissolving Pulp Committee for 1960. Mr. Charbonnier commented on the subject: In the past the Alkaline Pulping Committee tried to avoid joint meetings because the other groups might decide at a later date to split off leaving us weaker. Occasional joint meetings, however, are acceptable. This matter was tabled for a later date.

In a discussion of the proposition to make the Alkaline Pulping fall meeting a part of a large conference of Pulp Manufacturing Division:

1. D. Wetherhorn commented in opposition to concurrent meetings which deny an opportunity for a person attending to hear all papers presented.

2. The question was raised that with such a large meeting, only the larger centers could accommodate the group with the attendent possibility of the elimination of mill visits. Mr. Running also commented along the same line. Mr. Charbonnier moved that the Alkaline Pulping Committee remain as it is in regard to fall conferences. This was seconded by Mr. Woodside, who brought out in duscussion this would not preclude joint meetings. Also in some year it may be that one fall meeting would be waived in favor of a divisional meeting. The vote was unanimous in favor of the motion to hold Alkaline Pulping fall meetings in accordance with past practice.

3. Mr. Wetherhorn suggested we invite other groups to join us. After considerable discussion, it was decided not to bring this up at the Pulp Manufacturing Division meeting. Chairman Limerick attended the division meeting.

W. R. Stengle distributed copies of, and discussed, his financial report on the 12th Alkaline Pulping Conference at Hot Springs, Ark. last September. On motion by Mr. Bannister, seconded by Mr. Wetherhorn, the report was accepted; and a vote of thanks extended to Mr. Stengle and his committee for an excellent job.

Mr. Pineo asked if a saving was effected in printing by doing it locally. Mr. Teeple replied that by doing it in New York, last minute changes can be made, but the committee can do as it likes in the matter. Mr. Parrott asked if it was normal to pay expenses of some speakers and mentioned expenses of Mr. Zobel. Chairman Limerick agreed it was in special cases. Mr. Zobel made it clear his school did not have expense money available. Mr. Teeple said TAPPI Headquarters had no objection as long as it was budgeted.

Mr. Fuller reported on the New York program and gave credit to National TAPPI for the program arrangements.

Chairman Limerick discussed the general plans for the fall meeting in Jacksonville to be held Nov. 5 and 6, 1959. S. J. Kaptain, St. Regis Paper Co., Jacksonville, Fla., has agreed to act as local arrangements chairman.

Mr. Running reported 15 papers lined up for the program and prospects of a good meeting.

E. S. Rice reported on local arrangements completed at this time.

After some further discussion of the matter, the 14th Alkaline Pulping Conference was set for Portland for August 21 thru 24, 1960, in the Multnomah Hotel.

Mr. Wetherhorn reported for the testing subcommittee. It is working up information on a test for activity. The decision was made to continue and arrive at a proposal. A revised method for white and green liquor analysis was proposed. On motion by Mr. Pesch, seconded by Mr. Parrott, this revision was accepted and recommended to TAPPI. The matter of the Pascoe-Borlew method previously passed is to be cleared up.

(b) Mr. Borlew reported the sulphate cook and digester corrosion committee was still watching developments and had nothing definite to report.

(c) Mr. Gillespie reported for the Monograph Committee. The committee submitted an outline for the monograph on chemical recovery in the alkaline pulping process. The committee recommended that approval by the parent committee and by TAPPI be sought.

Mr. Borlew moved acceptance of the outline and the report; and that the Alkaline Pulping Committee approve and recommend to TAPPI. Mr. Wetherhorn seconded and the motion carried. Mr. Gillespie is to remain as chairman and chairman Limerick is to prepare a committee project proposal.

M. B. Pineo reported for the nominating committee, and nominated D. Wetherhorn as vice-chairman. Mr. Woodside moved the nominations be closed, Mr. Pesch seconded the motion, and it carried. Chairman Limerick instructed the secretary to cast an unanimous vote for Mr. Wetherhorn.

A motion was made, seconded, and carried to adjourn.

ROBERT R. FULLER, *Secretary*

Mechanical Pulping Committee

A meeting of the Mechanical Pulping Committee was held at TAPPI Headquarters, New York, N. Y., May 28, 1959.

Present were:

J. K. Kirkpatrick, *chairman,* Bowaters Southern Paper Corp.
S. R. Parsons, *first vice-chairman,* Consolidated Water Power & Paper Co.
M. S. Htoo, *second vice-chairman,* International Paper Co.
J. H. Perry, *secretary,* Norton Co.
J. H. White, Keyes Fibre Co.
R. V. Osborn, Great Northern Paper Co.
J. F. Jahne, Diamond Gardner Corp.
F. W. O'Neil, State University of New York College of Forestry
A. J. Winchester, TAPPI Headquarters
R. A. Joss, Technical Section, CPPA

Mr. Kirkpatrick, chairman, opened the meeting promptly at 9:00 o'clock and requested the secretary to read the minutes of the meeting of Feb. 25, 1959. The minutes were accepted as read. The chairman called upon Mr. White to give a report on the activities of the subcommittee on new methods of mechanical pulping. Mr. White submitted a

letter to committee personnel in which he pointed out that a progress report would necessarily be limited due to the fact that he had received insufficient response from members from whom he had requested suggestions as to the function of his committee. In this letter he also pointed out that this subcommittee had been designated erroneously in an article recently in *Tappi*. He also made some reference to the terminology for definition of mechanical pulping. It was also mentioned that he had received a note from M.S. Anderson in which Mr. Anderson agreed with the idea of a manual being produced which would describe all existing methods, which would be changed annually to incorporate new developments.

A motion was made by Mr. White that the subcommittee of which he is chairman be officially designated as Subcommittee on New Mechanical Pulping Methods. This motion was seconded by Mr. Parsons and carried.

Mr. White suggested that the Mechanical Pulping Committee definition of mechanical pulping be publicized so that it may be accepted generally by the industry.

The official definition of mechanical pulping as established by the committee in the August, 1957, minutes is as follows:

"Pulps produced on a pulpstone and also processes involving mechanical pulps produced by mechanical defibering of chips after pretreatment by means of either heat or chemicals, but not both; or without pretreatment."

Messrs. M. S. Anderson, J. P. Corbin, K. E. Pingrey, E. M. Jenkins, P. Bersano, and W. H. de Montmorency serve with Mr. White on this project.

Acting on Mr. Anderson's suggestion of a manual giving descriptions of the various processes of mechanical pulping, the motion was made that the subcommittee write a résumé containing the descriptions which will be published in the December issue of *Tappi*. In order that it be published in December, this has to be in the hands of the TAPPI Headquarters no later than November 10. Mr. White has agreed to prepare the résumé. This subcommittee has been assigned Project No. 779.

Project No. 637, Study of Groundwood Wet Web Strength—Maung Htoo, Chairman. Maung Htoo reported that he had referred his project to the Testing Division by means of a letter to Jack Winchester, along with further information which he had developed. A. J. Winchester will bring this to the attention of the proper committee within the Testing Division. The additional information which was developed by Maung Htoo will be sent to the committee members.

Project No. 678, Effect of Closed White Water System on Groundwood Brightness—F. W. O'Neil, Chairman. Mr. O'Neil reports that he and his staff have not had the opportunity to do much on this, but that he expected to carry on this project in the very near future.

Project No. 636, Effect of Underwater Storage of Pulpwood on Groundwood Brightness—Karl E. Pingrey, Chairman. J. F. Jahne, representing Mr. Pingrey, reported that sufficient wood had been recovered after the ice went out of the river to enable them to make another complete run. This will be done at the first opportunity.

Project No. 591, Groundwood Manual—E. H. Johnson, Chairman. S. R. Parsons reported that he and Mr. Yankowski had not met as previous minutes stated. However, he and Mr. Yankowski had each taken portions of the manual and are working on them to finalize it. These gentlemen are getting together on either June 5 or 6, to complete the work.

Project No. 680, A Survey of Groundwood Mill Process Instrumentation—J. K. Kirkpatrick, Chairman. Mr. Kirkpatrick reports that he has done some preliminary investigating but that he had nothing to report at this meeting. R. V. Osborn and Maung Htoo were appointed, along with W. H. McPherson, Jr., to serve with Mr. Kirkpatrick on this project.

J. K. Kirkpatrick mentioned that the committee was short two members due to resignations, and suggested T. H. LaHaise as a possible candidate, and referred this matter to S. R. Parsons, first vice-chairman, whose duties include maintenance of membership.

New Business

A motion was made by J. H. White, and seconded by F. W. O'Neil that a notice of committee meeting and a copy of the agenda for the meetings be sent to mill management as well as to committee members. This was carried. J. K. Kirkpatrick will follow through on this motion and in addition, as requested, the secretary will forward copies of the minutes to mill management. The members were asked to submit the names of their management of whom copies of agenda and minutes should be forwarded. Mr. Kirkpatrick will also write a letter to all members, setting forth the requisites for membership in the Mechanical Pulping Committee. Maung Htoo made a motion that committee meetings be held at mill locations, if possible, to enable members to visit mills at the same time as a meeting. It was pointed out that this practice is followed by the Canadian Technical Section Committee and the West Coast Mechanical Pulping Committee. This motion was seconded by J. H. White and carried.

It was decided that the next meeting will be held on October 5 and 6, and at the invitation of the secretary, will be in Worcester, Mass., affording members the opportunity of visiting Norton Co. where construction of pulpstones and abrasives can be observed. It was also suggested that this invitation be extended to the Canadian committee. The secretary is pleased to do this.

S. R. Parsons made a motion which was seconded by Mr. Maung Htoo and carried, in which it was requested that the secretary send a letter to the members at some time prior to the next meeting. This letter will request members to bring mill problems, so that they may be discussed during a bull session at the next meeting.

The committee decided that there would be a groundwood program at the next annual meeting in February, 1960. M. S. Htoo was appointed chairman and will be assisted by F. W. O'Neil, J. H. White, and Earl R. Schafer. It was felt that there would be sufficient material for at least a half day session.

The Fourth International Mechanical Pulping Conference

After much discussion, the dates of Sept. 19–21, 1961, were selected for the Fourth International Mechanical Pulping Conference and the Edgewater Beach Hotel in Chicago was selected as the site. This location is central to the groundwood industry in both the United States and Canada and would not represent a serious obstacle to those attending from abroad. A. J. Winchester telephoned the Edgewater Beach Hotel and confirmed the reservations for this time. R. A. Joss, representing both the CPPA Technical Section and the Canadian Mechanical Pulping Committee, was in accord with these arrangements.

Upon motion of J. H. White, which was seconded by Fred O'Neil, a subcommittee of three was appointed by the chairman to meet with the Canadian Groundwood Committee counterpart for the purpose of selecting the general committee for the Fourth International Mechanical Pulping Conference. The general committee, comprising a general chairman, program chairman, publicity chairman, and local arrangements chairman, will be selected by the Joint Subcommittee and then will be submitted to the respective groundwood committees for ratification no later than Aug. 1, 1959. This subcommittee is composed of J. K. Kirkpatrick, A. A. Yankowski, and J. H. Perry.

The meeting adjourned at 5:00 p.m.

J. H. Perry, *Secretary*

Training and Education Committee

The meeting of the Training and Education Committee was called to order in Parlor E, Hotel Commodore, New York, New York on Feb. 23, 1959, at 2:15 p.m. by the chairman, C. E. Libby.

The chairman opened the meeting with a request for nominations for a new chairman. John Lewis and Lyle Jenness were nominated. E. M. Engels moved the nominations be closed. The motion was seconded and passed. C. P. Donofrio moved that the man receiving the largest number of votes be elected chairman and the second nominee elected vice-chairman. E. F. Thode seconded the motion and the motion passed without dissent. The ballots were distributed by the secretary and after counting indicated Lyle Jenness was elected to the office of chairman and John Lewis to the office of vice-chairman for the coming year.

C. E. Libby stated to the committee the need for literature for distribution to attract high school graduates to seek opportunities in the paper industry. He called upon William Aiken, chairman of the Industrial Division of TAPPI to explain what steps had been taken by TAPPI in preparing various types of recruiting literature. He indicated President Harrison of TAPPI had charged the Operating Subcommittee of the Technical Operations Committee to study what could be done to recruit college graduates to the paper industry. The committee submitted a proposal to the Executive Committee of TAPPI for the preparation of suitable pamphlets for this purpose. The Executive Committee approved the expenditure of $6000 for this use. In addition, Mr. Aiken indicated that TAPPI was definitely interested in promoting the industry at the high school level, and he felt would support the publication directed toward the recruiting of high school students. It was stated that a pamphlet consisting of eight pages and cover and a printing of 50 to 60,000 copies might be appropriate. I. H. Isenberg indicated the availability of a pamphlet published by the National Lumber Manufacturers Association and suggested the correlation of the recruiting literature with the APPA Community Relations Program. Mr. Aiken stressed the need for a definite plan and layout before the committee approached the Executive Committee for financial support. It was suggested a subcommittee be appointed to study and plan a recruiting pamphlet aimed at the high school level.

Mr. Lewis brought before the committee a suggestion to study the possibilities of accreditation by TAPPI of the various pulp and paper programs in the colleges and universities of the United States. The suggestion was made for the chairman to prepare a memorandum for presentation to the Executive Committee of TAPPI to request the appointment of a committee to study the advisability of TAPPI accreditation.

P. S. Bolton, co-editor of the College Textbook reviewed the progress of the book's preparation. He stated that all but two chapters of Volume I had been received and were processed. Volume II of the book had been completed and shipped to the colleges as of February 20, he reported. Complimentary copies will be sent to all the authors as soon as the volumes are complete.

J. W. Field of the Association staff discussed various phases of the copyright problem, translations, and the permanent form of the publication. He stated that the charge of $10 per copy came within 25% of covering the costs of the initial printing. Various committee members expressed the desire of retaining the loose-leaf form of the original publication; so that new material could be added conveniently. A definite decision was delayed until more persons had used the textbook in classroom work. The committee also discussed the advisability of including problems within the text. Lyle Jenness reported that the Chemical Engineering Committee of TAPPI was actively assembling chemical engineering problems for use in engineering programs and suggested some might be suitable.

Myron Clark suggested that the committee might take some action to interest high school science clubs and science classes in the paper industry.

C. E. Brandon reported the national TAPPI office had prepared packets of material suitable for lecture presentation to high school students.

The committee adjourned at 3:35 p.m.

R. G. Hitchings, *Acting Secretary*

Training and Education Committee

Converting and Consuming Division

A meeting of the officers of the Converting and Consuming Division was held at the Commodore Hotel in New York, on Monday, Feb. 23, 1959, at 4:00 p.m. The following individuals were present:

K. W. Britt, Scott Paper Co., *Chairman,* Wet Strength Committee
W. L. Hardy, International Minerals & Chemicals Corp., *Chairman,* 1959 Plastics Conference
C. S. Maxwell, American Cyanamid Co., *Secretary,* Wet Strength Committee
W. E. Morris, Allied Chemical Corp., *Secretary,* Plastics Laminate Committee
W. A. Schenck, Riegel Paper Corp., *Past Chairman,* Plastics Committee
C. J. Seiler, Behr Manning Co., *Vice-Chairman,* Plastics Laminates Committee
H. A. Spencer, Knowlton Brothers Co., *Chairman,* Plastics Laminates Committee
W. N. Stickel, Texon, Inc., *Chairman,* Plastics Committee
R. C. Sturken, Frank W. Egan & Co., *Chairman,* Converting and Consuming Division
A. J. Winchester, TAPPI

The 1959 Plastics Conference was reviewed by Walter L. Hardy, chairman. This conference is to be held at the Edgewater Beach Hotel in Chicago on Sept. 21 through 23, 1959. The program is to include a session on Plastic Fundamentals, Production of Plastic Paper Materials, Plastic Laminates, and a Panel on Converting. Several plant visits are under consideration.

The Wet Strength Committee reported that it is holding a conference jointly with the Fundamental Research Committee of TAPPI, at The Institute of Paper Chemistry in Appleton, Wis., on Sept. 9 through 11, 1959.

Plans for the 1960 Plastics Conference were reviewed and finalized. It was decided that this would be held at the Hotel Syracuse in Syracuse, N. Y., from Oct. 17–19, 1960. A trip to the laboratory of the State College of Forestry at Syracuse is contemplated in conjunction with the conference. Definite hotel reservations will be made by A. J. Winchester of TAPPI.

The Wet Strength and Interfiber Bonding Committee was invited to participate in the 1960 Plastics Conference, so that all committees of the Converting and Consuming Divisions would be represented. At a subsequent meeting of the Wet Strength and Interfibre Bonding Committee, it was agreed that they would accept this invitation and would participate in one session of this conference.

Locations for the 1961 Plastics Conference were discussed and it was agreed that a resort hotel in the Indiana or Ohio area would be the most desirable location. A. J. Winchester and R. C. Sturken are to investigate a possible site in this area.

H. A. Spencer reported that J. C. Pitzer of Formica Corp. would be unable to continue as the representative of the TAPPI Plastics Laminates Committee on the joint ASTM Committee. Mr. Spencer advised that he himself would be able to represent TAPPI on this committee for the coming year.

The question of the annual reports of the committees and the divisions, was discussed. A. J. Winchester pointed out that the TAPPI regulations call for an annual report to be submitted by each committee to the division chairman by January 1, and that the division chairman's report should be submitted to TAPPI headquarters by February 1. It was agreed that the division would follow this schedule in the future.

It was announced that Joseph C. Pullman of American Cyanamid Co., had been appointed vice-chairman of the Converting and Consuming Division. Mr. Pullman had formerly served as chairman of the TAPPI Plastics Committee and is a member of the TAPPI Editorial Board.

R. C. Sturken, *Chairman*

Coating Committee

A meeting of the *Coating Committee* was held during the *10th Coating Conference, May 25, 1959,* at the *Hotel Statler-Hilton, Boston Mass.*

Minutes of the meeting of Feb. 23, 1959 (New York), were approved with minor corrections as suggested by Mr. Willets.

Research Grant, Project No. 536 (J. T. Loomer)

Glen Hemstock reported on status of the project and invited suggestions and participation. He described in detail the approaches that are planned in studying the rheological properties of coating compositions. Professor Lyle Jenness also invited suggestions from members of the committee in connection with the work of the University of Maine.

Mr. Loomer suggested that Mr. Hemstock notify all members of the Coating Committee.

Monographs (C. G. Landes)

Lloyd Silvernail reported that the Protein and Synthetic Adhesives Monograph will be completed early this year. Seven chapters are ready for publication, two are being proofread, and one will be ready for proofreading by the end of May (Project No. 569).

A report was read by W. E. Sowerbutts concerning the status of Project No. 303, the monograph on coating additives. Very few volunteers have come forward for this monograph and, because of this and other complications, assistance was requested from the council. Mr. Landes stated that steps would be taken to seek additional men for the project subcommittee.

G. L. Booth distributed copies of his report on activities of the subcommittee on a new project, a revised monograph tentatively entitled "Machinery and Methods for Paper Coating Colors." The project has been well organized by Mr. Booth and Vice-Chairman Nathan Millman, and it is hoped to publish the monograph by late 1960 or early 1961. This project will be formally set up with TAPPI by Mr. Landes.

Mr. Loomer suggested the possibility of preparation of a monograph on the elementary aspects of coating rheology. Mr. Hemstock voiced the opinion that it would be better to rely on the general textbooks on rheology. Mr Frankevicz suggested that a bibliography on rheology be published by the committee, particularly with beginners in mind.

Committee Projects (J. E. Wilber)

Project No. 600, Particle Packing. Reported by Mr. Hagemeyer. Technical report to be given later in the program. It was noted by Mr. Wilber that this work has been done voluntarily by the subcommittee members, with no use of TAPPI funds. Mr. Hagemeyer read a report by subcommittee chairman, J. V. Robinson, suggesting the desirability of extending the research work if at all possible.

New Projects, J. Frankevicz. Mr. Frankevicz reported that he has added several new members, including Messrs. Ritson, Neubauer, Whelpton, and Glassman. He also asked for a member from the board coating industry. He suggested: (1) more cooperation with paint rheologists, (2) establish liason with ceramic industry on rheology, and (3) extension of cooperation with ASTM on subjects such as the properties of latices.

Mr. Wilber asked for suggestions on how to collaborate with the paint industry. Messrs. Hale and Brooks offered to assist in establishing suitable contacts. Mr. Wilbur asked Mr. Frankevicz to contact Messrs. Hale and Brooks, and bring in specific recommendations for collaboration with the paint industry.

Mr. Willetts asked about what committees had been set up as a result of Mr. Kesler's committee report (refer to Pigment Studies Subcommittee of the Coating Committee, April 6th report by Mr. Willetts). He was anxious that no overlapping occur.

Mid-Year Coating Committee

Mr. Loomer reviewed past action on this point and asked for further comments. A show of hands indicated only five in favor of a midyear meeting. Mr. Hughson brought up the difficulty in justifying attendance at such a meeting. It was agreed that no further consideration would be given to this idea for the near future.

1960 Coating Conference (R. W. Hagemeyer)

Mr. Hagemeyer reported the following appointments to the Conference Committee:

R. W. Hagemeyer, *General Chairman*
W. E. Welliver, *Publicity Chairman*
C. W. Cairns, *Local Arrangements Chairman*
R. W. Martinek, *Technical Program Chairman*

The theme of the meeting will be "Coating for Printing." It will be a joint conference with the Graphic Arts Committee. Program suggestions and papers were requested. Mr. Loomer requested particularly papers from coating mills.

Mr. Loomer suggested holding the Coating Committee meeting at the 1960 Conference on Sunday preceding the Conference. A show of hands was 20 in favor and 30 against. However, this will be reconsidered at the February Coating Committee meeting.

Future Conferences (Mr. Loomer)

Conferences are scheduled through 1963 as follows:

1960—Edgewater Beach Hotel, Chicago, Ill., May 23–25
1961—Statler-Hilton Hotel, Buffalo, N. Y., May 15–17
1962—Netherland-Hilton Hotel, Cincinnati, Ohio, May 14–16
1963—French Lick-Sheraton, French Lick, Ind., May 19–22

New Orleans is being considered for 1964.

Data Sheets (Mr. Kaulakis)

Louis Georgevits has been appointed to head up this activity, and he requested that all committee members cooperate in sending in data sheets on coating subjects.

Liason Reports (Mr. Landes)

TAPPI Joint Advisory Committee on Printability. Mr. Landes reported that this committee played a part in initiating several research projects now being conducted by various TAPPI committees. The Joint Advisory Committee itself has been inactive for several years, so no liason is now required. If it is reactivated at any time, the Coating Committee will be invited to appoint a representative.

ASTM Committee D-25, Test Methods for Casein and Soya Protein. A report by J. C. Rice was read. Proposed methods have been circulated for comments to all members of the Coating Committee.

Routine Control Methods (J. R. Gunning) Mr. Gunning reported that several control methods have been published recently in *Tappi.* He requested suggestions on how to encourage mills to submit new control methods. It was brought out that even controversial procedures can be published; they need not be official or standard.

Committee Membership (C. G. Landes)

Requirements for committee membership were reviewed. It was stressed that all Coating Committee members should write the Coating Committee secretary when they are unable to attend a Committee meeting. Nonexcused absence from two consecutive meetings is cause for dropping a person from the Committee rolls.

A current membership list will be mailed out shortly.

Comments on Current Conference (F. Kaulakis)

Mr. Kaulakis mentioned the hard work done by members of his Conference Committee, especially program chairman Mr. Hagemeyer and local arrangements chairman Mr. Kilty.

New Business

Pigments Studies Subcommittee (W. R. Willets). Refer to report dated April 6, 1959.

Mr. Loomer introduced Harris Ware (representing Divisional Chairman Trelfa), who commented on the fact that the "Council" setup has provided a continuing source of new blood for conducting the Coating Committee activities. He complimented all members of the Coating Committee on their attendance and interest.

C. G. Landes, *Secretary*

Graphic Arts Committee

A meeting of the Graphic Arts Committee was held in the Commodore Hotel, New York, N. Y., Monday, Feb. 23, 1959.

The following members and guests were present:

C. A. Morton, *Chairman,* Kimberly-Clark Corp., Chicago, Ill.
M. C. Rogers, *Secretary,* Chicago Paper Testing Lab., Flossmoor, Ill.
R. H. Simmons, Bureau of Printing & Engraving, Washington, D. C.
R. J. LaFond, St. Regis Paper Company, Deferest, N. Y.
R. W. Roberts, Marathon Div., American Can Co., Menasha, Wis.
Frank B. Lincoln, Jr., Time, Inc., Springdale, Conn.
R. L. Hooper, Ecusta Div., Olin Mathieson, Pisgah Forest, N. C.
W. C. Walker, W. Va. Pulp & Paper Co., Williamsburg, Pa.
Jacqueline Fetsko, Lehigh University, Bethlehem, Pa.
L. Ed. DeLauter, W. Va. Pulp & Paper Co., Williamsburg, Pa.
C. H. Borchers, Litho. Tech. Foundation, Chicago 16, Ill.
J. H. Wing, Continental Can Co., Uncasville, Conn.
Edw. J. Heiser, Dow Chemical Co., Midland, Mich.
Riley M. Bates, Print Magazine, New York, N. Y.
Fred Weymouth, Interchemical Corp., New York, N. Y.
Victor J. Porth, Jr., Interchemical Corp., New York, N. Y.
D. J. Bernard, Interchemical Corp., New York, N. Y.
H. O. Ware, Past *Chairman,* Coating Division TAPPI
R. T. Trelfa, *Chairman,* Coating Division TAPPI
M. S. Kantrowitz, U. S. Govt. Printing Office, Washington, D. C.
T. E. Irwin, Oxford Paper Co., Chicago, Ill.
George Meade II, Consolidated Water Power & Paper Co., Wis. Rapids, Wis.
Alex Glassman, R. R. Donnelley & Sons, Co., Chicago, Ill.
R. J. Quint, Oxford Paper Co., Rumford, Me.
J. Parrott, Champion Paper & Fibre Co., Pasadena, Tex.
T. Lashof, National Bureau of Standards, Washington, D. C.
Clyde Eckhart, Ohio Boxboard Co., Rittman, Ohio
W. C. Chapman, Union Bag-Camp Paper Corp., Savannah, Ga.
George L. Booth, Black-Clawson Co., Fulton, N. Y.
Arthur E. Jones, Oxford Paper Co., Rumford, Me.
H. Ward Verseput, Robert Gair Co., Uncasville, Conn.
L. K. Burnett, Ohio Boxboard Co., Rittman, Ohio

Minutes of the Feb. 19, 1958, meeting were read and approved. The chairman recognized H. O. Ware, who introduced R. T. Trelfa as the new chairman of the Coating and Graphic Arts Division. Mr. Ware further reported that of 22 projects prepared by various division committees, 13 had been approved as research projects.

Group chairmen reported on their assignments:

Miss Jacqueline Fetsko (Papers) reported on the status of publication of the papers presented at the 1958 Graphic Arts session, and the papers presented at the Savannah Testing Conference in October. Plans are under way to participate in the program of the *1960* Coating Conference The 1959 Coating Conference program is already completed.

A. Glassman (Surface Strength). Detailed reports of the project carried on at Lehigh University have been received and studied by the members of the group (Messrs. Walker, Roehr, and Glassman). Summary reports have been mailed to Graphic Arts Committee members for comment, and further information was to be presented at the Graphic Arts Session of this annual meeting. The work shows large variations in day-to-day results of individual instruments, and between instruments. The best that can be said is that the instruments rank the sample papers in their "popular" rank order.

Good cooperation between the project and the instrument suppliers has existed. Additional funds ($10,000) to continue the project have been requested from TAPPI. Action is expected in April.

There was considerable discussion of the results of this project and their effects on the attitudes of the users toward the instruments tested. Some of the comments related to the false conclusions which might be drawn from the incomplete work leaving the instrument users in doubt about apparatus for which they have much use and application.

Glassman commented that some of the problems may relate to the small differences between the sample papers. He added, too, that in spite of the results reported, each laboratory appeared to group the samples in essentially the same order.

Oppen (Proof Press Techniques) reported by letter that the limited group size had reduced its activities, and he emphasized the need for definitions for graphic arts terms and suggested appointment of nomenclature committee. W. Walker reviewed the Research and Engineering Council of the Graphic Arts activities in that direction, and stated that a graphic arts dictionary had already had very much work done on it, but limited funds had caused it to be discontinued.

W. Walker and C. Morton are to review the membership in this group with the objective of improving its functioning. New members include W. C. Walker and H. Ward Verseput.

W. Walker (Ink Receptivity) reported a program for research study had been submitted for consideration and would be revised and presented in April, 1959. In connection with

Walker's request for approval of his program by other interested groups, it was suggested that he avoid the need for approvals because of the delays involved.

M. Rogers (*Letterpress Monograph*) reported that preliminary outlines had been prepared. Authors for sections of the monograph were suggested, and included Messrs. Bates, Paul, Larocque, Lincoln, Wing, and Calgren.

W. Verseput (*TAPPI Project 139*) reported on the first program which was completed in October. The second section will be reported at the Graphic Arts session this meeting. Application has been made of the Wink-Van der Akker ink spreading techniques. He reported that funds for the work are exhausted and a new request is to be made for $7000 from TAPPI. The Institute of Paper Chemistry has offered to add an additional $5000. Other work involving about $10,000 of TAPPI funds for work on compression properties will not be requested at this time.

T. Lashof (*Air Leak Smoothness*) reported that laboratory differences in the round robin tests were very great. The subcommittee is now attempting to determine the causes for these differences, and it may be necessary to use standard samples to check method reliability.

Old Business. It was reported that the subcommittee for *Project 346*—"Proof Press and Printing Quality" is interested in suggesting a method for standardized proof press operations. *Project 347*—"Proficorder"—The Institute of Paper Chemistry has requested that the supplier carry on work cooperatively with them. A. Glassman suggested that the R. R. Donnelley instrument might be available for some assistance to this committee. *Project 572*—Definition of terms is continuing. *Project 573*—of surface strength is continuing. *Project 616*—The survey of proof press techniques is temporarily inactive, but will continue. *Project 617*—Smoothness and compressibility is being handled by another group, and will be dropped from this committee's activities.

In the discussion of committee activities, it was suggested that the routine proof press tests should be a function of the special committee, headed by Oppen. Weymouth commented that there are so many variables involved in proof press tests, such as paper, ink, screen rulings, and whether the work is for packaging, publication, or other commercial work that the standardization on a single proof press test seems unlikely. Further discussion indicated that committee members were interested in a compilation of proof press tests as they are used. It was moved by J. Wing, seconded by W. Walker that "A committee be appointed to assemble and publish proof press techniques in use for routine control." Motion to amend was made by M. Kantrowitz as follows: "Move that the Printability Subcommittee be requested to study and survey the area of proof press techniques with the specific aim of recommending routine control methods for printability evaluation." This was carried. The original motion was then also carried.

It was moved by W. Walker "that the definitions of terms in the field of printability already developed by the special committee on printability be submitted to this committee for approval as a special one toward their publication." Motion was carried.

C. H. Borchers discussed developments of interest to Graphic Arts which were taking place at the Lithographic Technical Foundation and in the lithographic industry.

M. C. Rogers, *Secretary*

Corrugated Containers Engineering Committee

The *TAPPI* Corrugated Containers Engineering Committee met in Room 1048, Hotel Commondore, New York on Feb. 24, 1959.

Present were:

R. C. Hutcheson, *Chairman*, Union Bag-Camp Paper Corp., Savannah, Ga.
A. Richardson, *Vice-Chairman*, Gaylord Container Corp., St. Louis, Mo.
R. C. Berger, *Secretary*, General Electric Co., Schenectady, N. Y.

Members

W. E. I. Davies, International Paper Co., Whippany, N. J.
D. C. Ellinger, Downing Box Co., Milwaukee, Wis.
A. H. Fiedler, Deeter & Knabe, Pittsburgh, Pa.
O. Kleinman, S & S Corrugated Paper Machine Co., Brooklyn, N. Y.
J. E. Knecht, Bathurst Containers Ltd., Whitby, Ontario
L. W. Kutz, Reliance Electric & Engrg. Co., Bala Cynwyd, Pa.
R. D. Merrill, Stone Container Corp., Chicago, Ill.
H. W. Moser, S. M. Langston Co., Camden, N. J.
R. L. Newton, Owens-Illinois Glass Co., Toledo, Ohio
C. D. Nitchie, Koppers Co., F. X. Hooper Div., Glenarm, Md.
C. H. Robson, Lanzit Corrugated Box Co., Chicago, Ill.
F. E. Westfall, Lanzit Corrugated Box Co., Chicago, Ill.

Guests

O. J. Billman, Owens-Illinois Glass Co., Fairmont, W. Va.
J. M. Delfs, General Electric Co., Schenectady, N. Y.
M. R. Hamarat, Union Bag-Camp Paper Corp., Savannah, Ga.
M. M. Jenkins, Owens-Illinois Glass Co., Bridgeton, N. J.
W. B. Lincoln, Jr., Iland Container Corp., Indianapolis, Ind.
F. C. Masal, Owens-Illinois Glass Co., Toledo, Ohio
H. T. Scordas, Union Bag-Camp Paper Corp., Savannah, Ga.
R. O. Spalding, Owens-Illinois Glass Co., Toledo, Ohio
J. G. Turk, Glass Container Institute, Lansing, Mich.

The meeting was called to order at 9:20 a.m. by retiring Chairman Merrill. Committee members and guests introduced themselves. The meeting was then turned over to incoming Chairman Hutcheson.

High lights of the minutes of the Cincinnati meeting were read and approved. It was brought to the attention of the secretary that Messrs. Nitchie, Richardson, Moser, and Kleinman did not receive copies of these minutes. H. O. Teeple will be asked to correct this deficiency. (Note: Copies have been distributed.)

The secretary was instructed to make up a new list of committee members including their title, company, and complete mailing list. This is to be distributed to all committee members and to H. O. Teeple, TAPPI Headquarters.

Chairman Hutcheson introduced R. O. Spalding of Owens-Illinois Glass Co. who made a formal presentation covering some rather extensive research and development work on a new C flute design. High lights of Mr. Spalding's paper follow:

Corrugations per foot; 39 instead of standard 42.
Flute height: 0.142 in.
Tests show following general performance of 39-C flute over 42-C:
10% improvement in flat crush
4% reduction in take up factor
5% reduction in starch consumption

Corrugated Containers Engineering Committee (New York meeting)

Mr. Spalding concluded his remarks by stating that detailed information will soon be published giving complete details on the mathematical research which was confirmed by production tests. This presentation stimulated a rather lively discussion.

Mr. Richardson, chairman of the Roll Standards Subcommittee, thanked Mr. Spalding and the Owens Illinois Glass Co. for their contribution to the corrugated container industry.

Motion by Mr. Richardson, seconded by Mr. Nitchie that the roll standards previously submitted to the Steering Committee be withdrawn and that further consideration be given to these standards in light of Mr. Spalding's presentation. Motion carried.

Report on Waste Handling

Mr. Krecht's report had been mailed to all committee members prior to the New York meeting.

In answer to Chairman Hutcheson's question, Mr. Knecht reported that about 12 companies were included in his report, although all reports were not detailed and complete.

Mr. Richardson precipitated a discussion with regard to the proper dissemination of information which has been documented by our subcommitees. Several suggestions were made such as (a) publish in *Tappi*, (b) publish in trade journals, and (c) formal presentation at TAPPI meetings. Mr. Merrill was asked to refer this problem to the Steering Committee for a decision.

Report on High Temperature Belts

Due to Mr. Cassady's absence there was no formal report. A discussion revealed considerable interest in heat resistant belts and that some evidence indicating that increased life can be expected—anywhere from 9 months to 2 years.

Chairman Hutcheson will contact Mr. Cassady and specifically request a formal report be submitted at the New Orleans meeting. Mr Hutcheson will also suggest that Mr. Cassady get in touch with Messrs. Knecht, Richardson, Westfall, and Davies, all of whom seem to have had extensive experience with high temperature belts on corrugators.

Report on Box Plant Design

Mr. Fiedler indicated that he had not received additional questionnaires since the Cincinnati meeting. Several members indicated that they will be sending in answers to the questionnaire in the very near future.

Mr. Fiedler and his committee hope to receive the outstanding questionnaries and be in a position to present an interim report at the Pittsburgh meeting in May. This will include a statement of the objective and an outline of the job to be done. At present, Mr. Fiedler hopes to divide the work into areas such as (a) trim storage, (b) materials handling systems, (c) starch systems, and (d) plant construction. The committee will be asked for their comments before any extensive work is started.

Determination of Take-Up Ratios

H. W. Moser of S. M. Langston Co. distributed a prepared write-up on various methods for measuring take-up ratios and suggested that it might form a basis for discussion.

Mr. Moser indicated that there is no TAPPI standard established for the measurement of take-up ratio and many of the suggested methods are subject to criticism. It should be the objective of this committee to work out a standard method for measuring the take-up ratio in corrugating medium.

Messrs. Nitchie and Merrill outlined similar methods using revolution counters connected to wheels of known circumference together with built in timers or stopwatches. Various methods for reducing both instrument errors and human errors were discussed in detail.

It was pointed out that moisture content will affect results when using the "soaking method." It was the consensus of the committee that the soaking method should not be considered.

It was concluded that this problem warranted the formation of a subcommittee to study and recommend a standard method for measuring the take-up ratio in corrugating medium that would be accurate and used conveniently in the field. Accordingly, F. E. Westfall accepted Chairman Hutcheson's appointment to act as chairman of this subcommittee. Mr. Moser agreed to work with Mr. Westfall.

Power Requirements for Paper Converting Machinery

R. C. Berger presented a proposition for committee consideration relative to power requirements for corrugators, printer slotters, embossers, laminators, box folders, box gluers, and sheeters. Since TAPPI DATA Sheet 145G provides guides for paper machine power requirements and since TAPPI Project No. 368 involves the power requirements for supercalenders, it seems appropriate to consider the documentation of similar data in the paper converting area. After a short discussion it was decided to table this suggestion until the next committee meeting.

Planning for the New Orleans Meeting

Chairman Hutcheson reported that D. B. Phillips of Longview Fibre has agreed to present a paper on "Warp Control."

Chairman Hutcheson also reported that F. Glass of Green Bay Box was not in a position to present a story on web alignment. It was suggested that P. Ziegler of Fort Wayne Corrugated Paper Co., Hartford City, Inc., might be a candidate for this presentation. Mr. Hutcheson will contact.

C. Lawler of Union Bag-Camp Paper Corp. has accepted the assignment to present a paper covering the general subject of "Waste Areas in Box Plants, New Developments and Unsolved Problems."

After considerable discussion relative to a paper on "Knife Accuracy" it was decided that Maxwell Brothers be asked to present an objective paper on their experience with the Hooper knife together with statistical data on their experience to date. Since our meeting Chairman Hutcheson contacted Mr. Wright of Maxwell Brothers who agreed to provide a speaker for the New Orleans meeting.

If a fifth paper is needed for the New Orleans meeting or if an alternate paper is needed on short notice it was suggested that Mr. Spalding of Owens Illinois Glass Company be approached to give a more complete presentation on the 39-C flute design.

Date and Place for Spring Meeting

The spring meeting of the Corrugated Containers Engineering Committee will be held at the Pittsburgh Airport Hotel on Wednesday, May 20, 1959, at 9:00 a.m. H. O. Teeple will be asked to make arrangements for a meeting room, however, committee members will be responsible for making their own hotel reservations.

There being no further business, the meeting was adjourned at 12:30 p.m.

R. C. Berger, *Secretary*

Pittsburgh Meeting

The Engineering Committee met in Room M129, Pittsburgh Airport Hotel, Pittsburgh, Pa., on May 20, 1959.

Present were:

R. C. Berger, General Electric Co., Schenectady, N. Y.
W. E. I. Davies, International Paper Co., Whippany, N. J.
D. C. Ellinger, Downing Box Co., Milwaukee, Wis.
A. H. Fiedler, Deeter and Knabe, Pittsburgh, Pa.
R. C. Hutcheson, Union Bag-Camp Paper Corp., Atlanta, Ga.
Oscar Kleinman, S and S Corrugated Paper Machine Co., Brooklyn, N. Y.
J. E. Knecht, Bathurst Containers Ltd., Whitby, Ont., Canada

L. W. Kutz, Reliance Electric & Engineering Co., Bala Cynwyd Pa.
R. D. Merrill, Stone Container Corp., Chicago, Ill.
H. W. Moser, Samuel M. Langston Co., Camden, N. J.
R. L. Newton, Owens-Illinois Glass Co., Toledo, Ohio
C. D. Nitchie, Koppers Co., Inc., Glenarm, Md.
A. Richardson, Gaylord Container Corp., St. Louis, Mo.
C. H. Robson, Lanzit Corrugated Box Co., Chicago, Ill.
H. F. Thorn, Koppers Co., Inc., Bordentown, N. J.
F. E. Westfall, Lanzit Corrugated Box Co., Chicago, Ill.
H. O. Teeple, TAPPI, New York, N. Y.

The meeting was called to order at 9:15 a.m. by Chairman Hutcheson. The minutes of the New York meeting were read and approved.

Chairman Hutcheson introduced Harry F. Thorn, Koppers Co., Inc., a new member who has joined the Engineering Committee. The membership is now divided as follows:

Box Plant Operators	11
Machinery manufacturers	7
Architect engineers	1
Total	19

Discussion of New Orleans Papers

In an effort to provide an outstanding technical session at New Orleans, the Executive Committee suggests that the expected content of each paper be thoroughly discussed by the committee. Suggestions that result from this discussion should provide the speaker with a feel for the knowledgeability of his audience and the type of questions that he may be expected to answer.

Accordingly, Chairman Hutcheson appointed two men to take comprehensive notes of the discussion of each paper. These men, one representing the operator's viewpoint and the other representing the machinery manufacturer's viewpoint, will correlate, digest, and edit these notes for the benefit of the speakers. Final notes should be sent to Hutcheson who in turn will distribute them to the respective speakers with a covering letter. The following men were appointed by Hutcheson.

A. "Warp Control with Belt Driers," W. E. I. Davies, International Paper Co., and Oscar Kleinman, S and S Corrugated Paper Machine Co.
B. "Waste Reduction Possibilities through Engineering," J. E. Knecht, Bathurst Containers, Ltd. and H. W. Moser, S. M. Langston Co.
C. "Dorr Rapi-Die Register," D. C. Ellinger, Downing Box Co. (the author) and C. D. Nitchie, Koppers Co., Inc.
D. "Mechanical-Hydraulic Automatic Slitter Control," A. Richardson, Gaylord Container Corp. and H. F. Thorn, Koppers Co., Inc.

In an effort to create more interest in the engineering session at New Orleans, Chairman Hutcheson suggested that each member of the committee forward to him their thoughts with respect to more intriguing and/or thought provoking titles for each of the four papers that are presently planned. A total of four suggestions from each member (one for each paper) should provide a wealth of ideas for effective and outstanding titles.

It was further suggested that the usual question-and-answer period that traditionally follows the oral presentations be replaced by an informal panel to be made up of the speakers and one representative from each machinery builder who is presently represented on the Engineering Committee. Since Chairman Hutcheson is also the moderator of the session, it was suggested that he make whatever arrangements that he feels are necessary in this regard.

Corrugated Containers Engineering Committee (Pittsburgh meeting)

Take-up Ratio Project

Mr. Westfall made an informal oral report concerning his subcommittee's thoughts on the approach to this project. Mr. Westfall and Mr. Moser both feel that this is a most controversial subject due to the fact that there are so many variables that will influence the answer.

It was the feeling of this subcommittee, that although take-up ratio is directly influenced by flute contour; nevertheless, such factors as moisture content, temperature, humidity, and back tension should be considered in evaluating the method by which the measurement is made. Mr. Moser indicated that his company was working on a system for continuous monitoring of take-up ratio and that he would make this apparatus available for obtaining real engineering facts from the field. Mr. Westfall had no concrete information to present at this time; however he felt that his subcommittee needed some guidance as to how best proceed.

Mr. Richardson felt that the charter of this subcommittee rested in "the determination of a standard method for measuring take-up ratio" rather than "the determination of the factors and the degree to which these factors influence take-up ratio."

Although Mr. Westfall recognized that his subcommittee was charged with the search for a "standard ruler" nevertheless he felt that this "standard" should include the conditions by which the measurements are made. The primary objective is, of course, to determine if a "yardstick" or a "micrometer" is necessary for measurement.

It was agreed that this project might well be resolved into two approaches. Mr. Westfall and Mr. Moser will attempt to present an interim report at the New Orleans meeting which will be supported by actual field data.

Standard Corrugating Rolls Project

Mr. Richardson suggested that this project be tabled unless and until more factual comparisons are available between 39-C and 42-C flutes. Although there are over 30 39-C rolls in service there is still not enough information to form a basis for comparison.

Box Plant Design Project

Mr. Fiedler stated that the original objective of this project was to provide a manual to be used as a guide in box plant design. At the moment he feels that little if any progress has been made since last February. Out of 11 questionnaires that were sent out only five have been returned and the information received so far is rather incomplete in some areas. As a result Mr. Fiedler felt that this project should be abandoned. If this is not possible then he felt that he should resign as chairman of the subcommittee.

After considerable discussion which included the concept of breaking the project down into smaller areas, such as building design, raw materials handling, roll storage area, etc., it was moved by Mr. Fiedler that the project be abandoned. Motion seconded by Mr. Davies. Motion carried.

Mr. Robson stated that the committee was indebted to Mr.

Fiedler for his efforts in preparing a most comprehensive questionnaire which in itself is an excellent guide for box plant design and as such can be used as a reminder for the various facets that must be considered. The Engineering Committee therefore thanked Mr. Fiedler for his time, his patience, and his splendid contribution.

There being no further business, the meeting was adjourned at 4:15 p.m.

R. C. Berger, *Secretary*

Corrugated Containers Production Committee

The Corrugated Containers Production Committee met Feb. 24, 1959, at the Hotel Commodore.

Present were:

D. Adams, St. Regis Paper Co.
L. F. Ashwood, Downing Box Co.
D. Blackburn, Martin Paper Products
R. H. Bowers, Connelly Containers, Inc.
R. W. Buttery, Bathurst Containers, Ltd.
R. W. Catzen, Baltimore Paper Box Co.
J. Clarke, Longview Fibre Co.
D. C. Dowd, Chas. Dowd Box Co., Inc.
W. J. Hurrell, Hinde & Dauch
B. Mendlin, Cornell Paper Products Co.
K. Provo, Gaylord Container Corp.
A. C. Reid, Pembroke Chook Mills
H. W. Wilson, Royal Container Co.

The nominating committee (Bowers and Adams) presented a slate of officers, who were subsequently elected: Ralph Buttery, chairman; Don Blackburn, vice-chairman; Loren Ashwood, secretary.

Project No. 695, Adhesive Factors

TAPPI allocated $500 with the understanding that 10 plants would each contribute $55 and file production and consumption reports so that this study may be conducted at the University of Manitoba,

Mr. Blackburn explained that currently the relative amounts of adhesive used for different flutes are not accurately known. Various factors are used by different companies with 1.33 for B flute based upon A flute being one of the more popular figures. From the work which has been done thus far at the University, the adhesive factors for one machine have been determined. It is expected that as a result of this project, factors will be developed for each of the machines participating. The information will allow breaking this down further by makes of machines and by areas of the country. It may not be possible to compare machines one with another, but a range of factors for all of the machines of each flute should be attained. Then after studying these machines, the subcommittee will be better able to determine the value of such data and the future course of action to be taken.

Because observations for at least 200 days production are needed for the accuracy desired, it was decided that the participating plants made up of companies represented by the members of the Production Committee should compile starch figures for March 1 through Dec, 31, 1959. The figures should be in pounds of dry starch so as not to be misled by differences in adhesive formulas used by various companies. It was pointed out further that it is not important whether the board produced is given in usable square footage or in total square footage just so long as each plant does it consistently thruout the reporting period. The condition or the adjustment of rolls might affect the results.

It was considered that the relative amounts of A, B, C, and DW flutes should not affect the accuracy. If it is possible to separate starch consumption by A flutes (columns 11–20, 21–30 in the chart which Mr. Blackburn distributed for reporting the information), this would be fine; otherwise the total used on the single-facers and on the double-facer will

Corrugated Containers Production Committee

suffice. It was decided that if some isolated flute runs only occasionally, the production, and consumption figures can be eliminated, or—if someone prefers to show these—they can be entered on a separate line. Waterproof adhesive figures should be omitted.

Signature numbers were assigned to individual plants and an explanation given as to how to fill out the reports. To be certain that everyone understands how to do this, entries for possibly a week should be prepared by each plant and sent to the following committee members. Messrs. Provo, Reid, Dowd, Clarke, and Ashwood should send their reports to Mr. Bowers, while Messrs. Buttery, Bowers, Catzen, Mendlin, and Wilson should send theirs to Mr. Dowd. Once everyone understands how to prepare the reports, these should be kept daily by member plants and then the entire record submitted to Mr. Blackburn early in January, 1960. Actual work cannot start until all the figures are available, so there is no need to send these in piecemeal.

Project No. 694, Supervision

Mr. Buttery distributed a preliminary summary of the questionnaire distributed by the subcommittee. After some discussion on the practicality of further exploring this subject or of periodically conducting a similar survey so as to detect trends, if there are any changes, it was decided to close the project and retain the information for reference for a possible future paper.

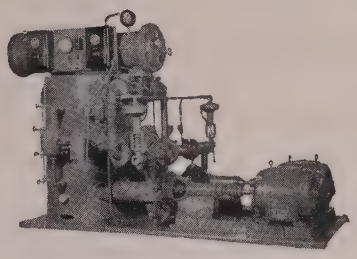
Supervisory Training

Mr. Ashwood reported on the results of a questionnaire prepared by the subcommittee and circulated among the members. Subjects necessary and subjects desirable to be covered in a training program were presented with the minimum discussion time recommended that should be devoted to each subject. High lights of individual training programs in various plants were covered. The subcommittee plans to prepare suggested study outlines for each of the necessary subjects to be used as a guide for anyone conducting such a program. These will also be circulated among the members for review before submitting to the committee as recommendations.

Labor Costs

Mr. Catzen reported that the Baltimore group met and decided that since so many plants use different crew sizes and assign various duties to individual crew members, it would be almost impossible to set up standards for the industry so do not feel that anything further can be done. After some discussion it was decided to drop the project.

The ONE Vacuum Pump Made for Paper Mills



The Stickle Vacuum Pump is made exclusively for paper mills. It's easy to service. All floats housed in exterior float boxes. Interchangeable, removable vacuum chamber is above tank. Inspection of ejector tubes may be made without moving motor, pump or piping. Uses standard horizontal motor and pump. For vacuums to 28". Built-in heat exchanger eliminates costly cooling equipment. Recirculation of hurling water condenses flash vapors and releases air and gases to atmosphere. No close clearances to cause wear. Performance guaranteed. Single and duplex models. Write for free literature.

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Moisture Control Systems
Dryer Drainage Systems
Feed Water Heaters
Vacuum Pumps
Micro Adjustable Orifices
Differential Control Valves

New Projects

Mr. Provo pointed out that the trend toward smoother surfaces on liner board is creating problems with machines using suction cup feeders. He suggested that a study be conducted to develop a test to measure the ability of board to be picked up by suction cups on automatic feeding equipment. No action was taken on this.

As a second project Mr. Provo suggested that a study be made of maintenance hours relative to the operating hours of major equipment. As a guide to the effectiveness of a maintenance program, breakdown hours could be considered. After some discussion, Mr. Catzen was appointed chairman with the instructions to determine if there is some way to measure the effectiveness of maintenance. He will work with the Baltimore group on this problem.

Mr. Blackburn suggested a project to consider standardizing on score allowances. He also suggested one on methods of printing to obtain high quality results what with the demand for this increasing regularly. No action was taken on either of these.

February Meeting

Mr. Buttery announced that Roscoe Bowers will be the general chairman of this meeting with the theme to be production. Suggestions for topics, moderators, and speakers were requested.

For topics, the group suggested papers on: Supervision (reference Project No. 694), score profiles, results and pay-out of using tapered speed drive relative to quality and glue consumption, waste, a series of short papers describing a specific idea found advantageous by individual plants, humidity control—water versus steam, job orders and specifications, scheduling machines, cutting die making, and a foreigner boxmaker's viewpoint of the American corrugated industry.

Suggested moderators and speakers included: David Dowd, John Means, Abe Lincoln, Lyn Falkner, Leon Hamilton, Wes Sykes, and Fritiz Theim.

Addition suggestions for speakers should be sent to Mr. Bower.

F. Ashwood, *Secretary*

Fourdrinier Committee

The spring meeting of the TAPPI Fourdrinier Committee was held at TAPPI Headquarters on Monday May 4, 1959. Chairman Kennedy called the meeting to order at 10:00 a.m. The following were in attendance:

F. L. Allen, Canadian International Paper Co., Montreal, P. Q.
J. Hannigan, International Paper Co., Corinth, N. Y.
P. H. Hershey, P. H. Glatfelter Co., Spring Grove, Pa.
R. W. Hisey, S. D. Warren Co., Cumberland Mills, Maine
W. H. Kennedy, Nekoosa-Edwards Paper Co., Port Edwards, Wis.
R. N. King, Nash Engineering Co., S. Norwalk, Conn.
S. J. Leishman. Provincial Paper Ltd., Thorold. Ont.
A. E. Levensailor. International Paper Co., Mobile. Ala.
H. H. Mullins, Olin Mathieson Chemical Corp., W. Monroe, La.
R. W. Peters, Eastman Kodak Co., Rochester, N. Y.
J. E. Schad, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.
H. S. Spencer, Howard Smith Paper Mills, Ltd., Beauharnois, Que., Can.
A. J. Winchester, TAPPI, New York, N. Y.

Mr. Allen, of the Charles Bruning Co. presented an illustrated talk on "The Optical Alignment of Rolls on the Fourdrinier," as a subject of possible interest to the committee. This technique was developed for the aircraft industry, as a substitute for mechanical means such as taut wires, squares, etc. The advantages are extreme precision and good speed. Mr. Allen stated this should result in increased wire life and decreased down time, especially on fast machines.

On Project No. 635, "The Problems of High Speed Machines Operation," Mr. Winchester will arrange transfer of the data to Mr. Levensailor for evaluation and examination before the fall meeting.

An animated discussion was held on the project suggested by Mr. Hershey, "Fine Paper Quality Control." This covers a very broad and important field. Included is the methods and organization of the control functions, stock preparation control, fourdrinier control, actual system design to allow control, etc. It was decided that the best attack on this subject would be to take a small bite at a time, keeping in mind the general area to be covered. Messrs. Hershey, Leishman, and Spencer were appointed as a subcommittee to study and progress the first section of this problem, which has been titled "Wet End Process Control." A report is to be presented at the fall meeting.

Project No. 734, Dandy Roll Practices. Mr. Hannegan reported that his subcommittee had prepared an outline for a questionaire. The outline was discussed and approved. Mr. Hannegan will consult the manufacturers of dandy rolls for comments, after which the final questionaire will be sent to Mr. Winchester.

Project No. 735, Shake. After some discussion this project was cancelled, as it was felt that this fit under the general heading of process control.

Project No. 737, Rope Carrier Practices. Mr. Mullins reported that he had obtained quite a bit of information and hoped for a paper on this subject to be presented in February at the National meeting.

In response to the questionaire on meeting sites, the choice of the committee is to meet near mills, providing good air transportation is available. Either Monday or Friday seemed suitable as a meeting day.

The fall meeting was tentatively scheduled for October 19 and 20 in Rochester.

The meeting was adjourned at 4:00 p.m.

R. W. Hisey, *Secretary*

ENGINEERING DIVISION

Committee Contributions

Questions and Answers

(*9*) *Question:* Fiber disposals from a kraft and newsprint mill are discharged into an open unlined ditch, then pumped to a thickener. The waste material is thickened to a range of 0.5 to 1% consistency and pumped into a storage settling basin where the heavier particles settle while the liquid is drained. In time, the reservoir becomes completely filled with pulp fibers and other rejects. After pumping into the reservoir is completed and most of the surface liquid is drained or evaporated, the consistency of the settled material then varies from 4 to 8%.

Could this material be thickened after leaving the clarifier to a consistency higher than 1% in order that it may be removed, or could the sludge be thickened after settling for cheaper removal from the reservoir?

If these rejects and fiber losses were recovered, is there any market value, or more applicable means than using as fill or fuel?

(*10*) *Question:* We employ a large number of chains in our operations for power transmission and conveyor purposes and are plagued with outages due to the breaking of the chains before a reasonable amount of wear has occurred. How do you calculate the safe working load for a chain?

(*10.1*) *Answer:* From a stress standpoint, a chain is a very complicated structure for the parts have relatively short spans combined with heavy cross sections and small deflections produce great stress. The stress in one part of the chain depends largely upon the deflection in another part and upon the fits that actually have been achieved in the chain fabrication rather than the dimensions placed upon the designers' drawings. Thus, rigorous calculation of stress in the parts is very difficult if at all possible. Because of this difficulty, chains are usually rated on the basis of their average ultimate breaking load in tension. The breaking load can be obtained in a tensile tester and then the ultimate load is divided by some factor derived from experience to convert the ultimate load to working load. These divisors are relatively large numbers varying from 6 to 18 or 20 depending upon the type and style of chain. Because they are relatively large, many designers become careless and feel that a precise estimate of the load to be imposed is not important and they mistakenly view these divisors as safety factors which they are not. These divisors are the numbers that are required to reduce the ultimate breaking stress in the chain to a value that will yield a reasonable life. Stresses higher than these place the material in the limited life range where the parts will fail from fatigue and if the loads have been slightly underestimated fractures will occur very quickly. It, therefore, is prudent to carefully determine the load to be carried by the chain, increase it by whatever safety factor seems to be indicated and then match this value to the working value of the chain obtained by dividing the ultimate breaking load by a suitable divisor—The divisor to be obtained by experience or from the recommendations of the chain manufacturers, who generally list the minimum recommendation in their catalogs. If you follow this procedure your chains should yield reasonable life and fail from wear instead of fatigue.

(*11*) *Question:* In the design of foundations for paper mill machinery: (a) How is the ratio of the mass of the foundation to the mass of the moving parts of the machine determined? (b) How is the foundation bolting proportioned to tie the machine to above determined mass?

(*12*) *Question:* There apparently are two schools of thought regarding the method by which sequential interlocking should be provided on a conveyor system, one method being the connecting of auxiliary contacts directly into the control circuit of adjacent starters. The other being the use of relays between the circuits of these starters. I would like to know the advantages and disadvantages of each method.

(*12.1*) *Answer:* We have established at our mill a practice of sequential interlocking that we feel is a good compromise between economy and maintenance simplification. In our opinion, all complex systems should be interlocked by means of relays and those relays should be located at one accessible point so that trouble shooting could easily be done from one spot. In addition to the use of relays, all wiring from remote photoelectric relays and limit switches is brought to this central point.

For simple two or three motor systems, the use of the interlock on the starters has been generally accepted, although it is not considered as desirable by the maintenance electricians. It is generally more difficult to maintain, service, or jumper the interlocks when they are mounted on starters. We object to a 440 volt shock from an interlock or any source when disconnect switches have been pulled. Most interlocks on starters are relatively inaccessible, and if as many as three interlocks are used on one starter, the entire movable element of the starter becomes a rather flimsy structure and, therefore, the chance of misalignment of contacts and burning of contacts becomes a problem. The starter has to be completely dismantled in order to change or repair the interlocks.

(*12.2*) *Answer:* Before going into the advantages and disadvantages of the relay method, it would be desirable to make a few generalizations. Both methods are used quite extensively, are reliable, and will accomplish the primary purpose of interlocking with equal effectiveness. The method to be used in most paper mills is to a large extent governed by what has already been established as standard practice; however, the trend in recent years appears to be toward the use of auxiliary relays. In cases where auxiliary relays are utilized, they are usually grouped together in a separate cabinet or compartment in a motor control center assigned exclusively for this purpose.

Advantages: (1) The use of relays permits placing orders for starting equipment without having to differentiate which starters must be equipped with auxiliary contacts and how many will be required on each. This is a considerable advantage when equipment has to be ordered before interlocking requirements are known. (2) Basic control wiring schemes for all starters can be standardized on regardless of the interlocking requirements. (3) The relays are standard and interchangeable and are all mounted in one enclosure simplifying maintenance or replacement. (4) Flexibility for future changes can be achieved by leaving space for additional relays. Relays can be readily added. (5) The various types of relays used for this purpose have at least two sets of contacts, thereby providing for spare contacts. (6) Relays permit the de-energizing of all wires in the starter compartment, when the line switch is open, thereby eliminating the hazard of having energized interlocking wires from some other source during periods of maintenance.

Disadvantages: (1) The relay method is more expensive; (2) involves additional space required for housing the relays; and (3) introduces the use of an additional coil which increases the number of possible sources of trouble.

(*14*) *Question:* Has a satisfactory means of "on the machine" roll grinding been developed?

(*15*) *Question:* With two different machine chest configurations, basis weight difficulties have been experienced with reduction of the chest level at the end of a run. With a vertical chest equipped with a vertical agitator the sheet runs light, indicating a loss of consistency. However, with a horizontal chest and archaic horizontal paddle agitation the sheet runs heavy, indicating higher effective consistency. No change or improvement in the latter was noted when a constant level overflow box was provided to give fixed head to the machine pump without regard to the depth of stock in the machine chest.

Since three types of pumps have been used, one of which has given identical performance with and without the constant level box, it seems that chest agitation rather than pump hydraulics is the problem. If so, are there any known chest and agitator configurations which will maintain homogeneity with the whole body of stock independently of its level?

Fourteenth

Engineering Conference

Penn-Sheraton Hotel, Pittsburgh, Pa.

Oct. 12–15, 1959

LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel, and Events

Golden Gate District (Robert M. True Memorial Lectures)

Robert M. True (deceased August, 1957) of General Dyestuff Corp., is credited with being the individual most active in promoting the organization of the Golden Gate District of

Wilfred Gallay delivering the Robert M. True Memorial Lecture

Pacific Section. He had served as secretary-treasurer of the Pacific Section for a period of 10 consecutive years (1943–52, inclusive) when his business headquarters was in Portland, Ore. He transferred to San Francisco in 1953.

In his memory, his company, General Dyestuff, and the Golden Gate District of the Pacific Section of TAPPI have established an annual lecture.

Walter Brandt, Wilfred Gallay, W. H. Kuefner, Mrs. Brandt, and Mrs. Robert M. True

Officers of the Gulf Coast District: D. F. Church, Crown Zellerbach Corp.; H. Rammer, Fibreboard Paper Products Corp.; H. W. Theller, Western-Waxide Div.; L. G. Maclise, Dow Chemical Co.; W. H. Kuefner, The Flintkote Co.; and Walter Brandt, General Dyestuff Co.

The first Robert M. True Memorial Lecture was given on May 19, 1959, when we were honored to have as the guest speaker, Wilfred Gallay, director of research, E. B. Eddy Co., Hull, Quebec.

Dr. Gallay gave a most interesting talk on "Strength Development," dealing with strength, resiliency, toughness, interweaving, and bonding of fibers. He followed this development from the chipper to the reel.

Elections were held on May 19, 1959, and the results were as follows:

Chairman: John Hartsock, International Paper Co.
Vice-Chairman: H. W. Theller, Western Waxide Div.
Secretary-Treasurer: H. A. Harvey, Penick and Ford Co. Ltd.
Recording Secretary: Walter Brandt, General Dyestuff Co.
Executive Committee: D. F. Church, Crown Zellerbach Corp.; L. G. Maclise, Dow Chemical Co.; H. Rammer, Fibreboard Products Co.

WAYNE H. KUEFNER, *Chairman*

Gulf Coast

The Charter Meeting of the Gulf Coast Section of TAPPI was held at the Jung Hotel in New Orleans, La., on May 15, 1959. Chairman J. E. Doyle presided at the meeting.

Following the social hour and dinner, a financial report on the year's operation of the Gulf Coast District was given by Treasurer N. M. LeMaistre. Next, the new officers for the coming year who had been proposed by the Nominating Committee at the Mobile meeting were elected. These are:

Chairman: William Elsevier, International Paper Co., Mobile, Ala.
Vice-Chairman: A. L. Hargraves, St. Joe Paper Co., Port St. Joe, Fla.
Secretary: N. M. LeMaistre, Container Corp. of America, Brewton, Ala.
Treasurer: M. T. Still, St. Regis Paper Co., Pensacola, Fla.

R. G. Macdonald (r) presents charter to Chairman J. Edward Doyle of the new Gulf Coast Section of the Technical Association of the Pulp and Paper Industry

Executive Committee: W. P. Shannon, St. Joe Paper Co.; J. E. Doyle, St. Regis Paper Co.; I. S. Walker, International Paper Co.; R. R. Fuller, Gulf States Paper Co.

Mr. Doyle then introduced R. G. Macdonald, who made the presentation of the Charter to the Gulf Coast Section of TAPPI.

Following this, Dick Chase, chairman of the Southeastern Section of TAPPI, spoke briefly.

Phil Nethercut, secretary-treasurer of National TAPPI, then presented a certificate of appreciation to retiring Chairman Ed Doyle.

J. P. Weidner, Technical Director of Container Corporation of America, then spoke on "Local Sections and TAPPI." This concluded the evening meeting.

On Saturday morning, William Elsevier served as moderator for a panel discussion of pulp and papr testing. Panel speakers were:

1. Earl Wright, Gulf States Paper Corp., Tuscaloosa, Ala.—"Comparison of a Mead Refiner with a Valley Beater in Laboratory Evaluation of Pulp Strength Qualities."
2. Frank D. Hall, Container Corp. of America, Brewton, Ala.—"Comparison of Wet Pad Colormaster Brightness with Dry Pad G.E. Brightness."
3. J. Burt Rhyne, International Paper Co., Panama City, Fla.—"Aluminum Foil Standardization of Mullen Testers."
4. Tom E. Linder, International Paper Co., Moss Point, Miss.—"Use of the I.G.T. Print Tester."
5. T. M. Cunningham, International Paper Co., Mobile, Ala.—"Betameter Chart and Plastic Rule Method as an Aid to Basic Weight Control."

Charter meeting of the Gulf Coast Section

6. Larry Clapp, St. Regis Paper Co., Pensacola, Fla.—"A Device for Measuring the Curling Tendency of Paperboard."
7. Leo Shealy, St. Joe Paper Co., Port St. Joe, Fla.—"The Use of Drainage Test with High Yield Kraft."

The speakers at the Saturday morning meeting were:

1. John Kressman, Continental Turpentine and Rosin Corp., Shamrock, Fla.—"Educate Your Guesses—with Statistics."
2. Clark E. Snook, Nopco Chemical Co., Newark, N. J.—"Recent Developments in Paper Coating Additives."

N. M. LeMaistre, *Recording Secretary*

Local Sections and TAPPI

J. P. Weidner

On this occasion, it seems proper that a brief look be taken at the past, present, and future of local sections.

TAPPI was organized on Feb. 19, 1915, as a section of the American Paper and Pulp Association. On Feb. 17, 1916, the articles of organization were revised and the Technical Association of the Pulp and Paper Industry emerged. In 1916 there were 270 members.

The Pacific Section was chartered in 1929, the Lake States Section in 1930, Delaware Valley and Kalamazzo Valley in 1931. At this point total TAPPI membership was 1198.

The next sections were New England in 1933, Ohio in 1935, Empire State in 1938, Maine-New Hampshire 1940, Chicago 1943. After 25 years TAPPI membership had grown to 1926 members.

No new local sections were formed during the war years and the next were Lake Erie 1951, Southeastern 1952, and Virginia-Carolina 1954.

The first Section to have Districts was Empire State, which developed districts because of the wide separation of areas where mills were located. Hence districts grew until there are now five in this Section.

The large growth in Districts occurred in 1956 and 1957 as follows: St. Louis District of Chicago, Golden Gate of Pacific, Eastern of New England, Indiana of Ohio, and Gulf Coast of Southeastern.

Our past secretary-treasurer, Mr. Macdonald, encouraged the formation and nutured the growth of the districts.

The present Executive Committee has appointed a committee to work with the Local Sections Operations Committee to suggest ways that TAPPI may assist the Local Sections and Districts, as regards uniformity of various practices such as dues, mailing notices, rebate policy on dues, financial assistance, geographical boundaries, publicity, and coverage of meetings.

You will be interested to know that the headquarters staff was asked to present a 5-year forecast on Local Sections and Districts. The prediction was an increase in Local Sections from 12 to 24 and a drop in the number of Districts from 9 to 2.

Mr. Harrison in his annual report to members stated that over 25% of our individual members and 45% of our corporate members are from outside the United States. During the past 5 years the TAPPI members in Europe have increased from 500 to 1000 members. The European Study Group was organized by TAPPI members. At this past annual meeting, the Executive Committee of TAPPI granted its first overseas Local Section charter to the Italian Section. These organizantions are a natural outgrowth of the large overseas membership.

All of the presentation of technical information is handled by individual TAPPI members either through committees or through local sections. Hence, the purpose of the organiza-

J. P. Weidner, Technical Director, Container Corp. of America, Brewton, Ala.

tion "organized for the search and dissemination of knowledge relating to pulp and paper" in the final analysis is an individual one. While committee activity is formalized, the success of the local section is less so and more dependent on individuals. Thus, it presents an ideal training ground for individuals to develop organizational skills as well as technical knowledge. Management supports the local sections mainly for these two reasons.

A proper question now is: Why should one join the local section of TAPPI? There are a variety of reasons—learning more about the industry and its problems, not only about the problems in one's own mill; the opportunity to enlarge ones circle of friends, for prestige and for the broadening of professional status. Certainly anyone who joins such a group soon feels the need to contribute to its welfare and growth. The effect is to realize more completely one's individual potential as we appreciate the challenge faced by the group.

Local TAPPI is especially pointed toward the junior men in our industry who may not have any other opportunity to attend TAPPI meetings or conferences. If TAPPI is to grow, it has the necessity of building the younger men to the point where they may actively take responsibilities in the parent organization.

One of the serious problems is to attract students in high schools and colleges to our industry. The local section is our most effective agency. To aid students who are interested in the pulp and paper industry the Executive Committee has established a special membership class for undergraduates. Annual dues are $4.00 and they may defer payment of their initiation fee until they have left school. For these dues the students receive the *Tappi* magazine but none of the other publications or services.

How have TAPPI members acted to further the educational opportunities of younger people?

Several local sections have sponsored educational programs at nearby high schools and colleges.

The TAPPI Training and Educational Committee has prepared a two-volume textbook for college use.

During the past 10 years TAPPI has financed more than 100 research studies, acquainting faculty members and students with industry problems.

The TAPPI Chemical Engineering Committee is preparing a textbook for college use on chemical engineering problems based on the pulp and paper industry.

TAPPI this year is preparing a brochure describing the opportunities for technical careers in our industry.

Professors from nearby colleges have been invited to functional conferences.

TAPPI and APPA are jointly sponsoring an educators conference at which chemistry and engineering professors and paper industry research leaders will discuss problems of the industry.

In the past few months TAPPI has sponsored paper talks at more than 50 ACS or AIChE Student Chapter meetings. These talks have been handled by TAPPI members from nearby mills. Many have been followed by mill tours.

Also countless members are serving on school boards, teaching at night, or working with organizations actively concerned with our educational system.

TAPPI has kits of speech materials, paper samples, and the like and is prepared to help in any way possible.

There is a challenge to all local sections to find in which direction it may be of the most service.

One of the most effective ways of building our younger people is with a meeting devoted to the presentation of papers by them and the giving of an award or awards to the best.

Now a word about the responsibilities of local sections to management.

For management to support the local sections, certain standards of technical information and professional and social standards are expected. If management sends people to meetings, it certainly expects that those attending will truly search for knowledge on a high professional level; that as far as seems wise, exchange general information and experience where such does not conflict with the interests of his employer.

Those in charge of the meetings have the responsibility for its technical excellence and the timeliness of the material presented.

These latter things are pointed out only because the granting of a charter by the Executive Committee is done only after serious consideration, and in the belief that the group will conduct itself in every way to uphold the high traditions of TAPPI. That this group has operated only 2 years as a District before being granted a charter as a Local Section certainly speaks highly for the people who have organized this group.

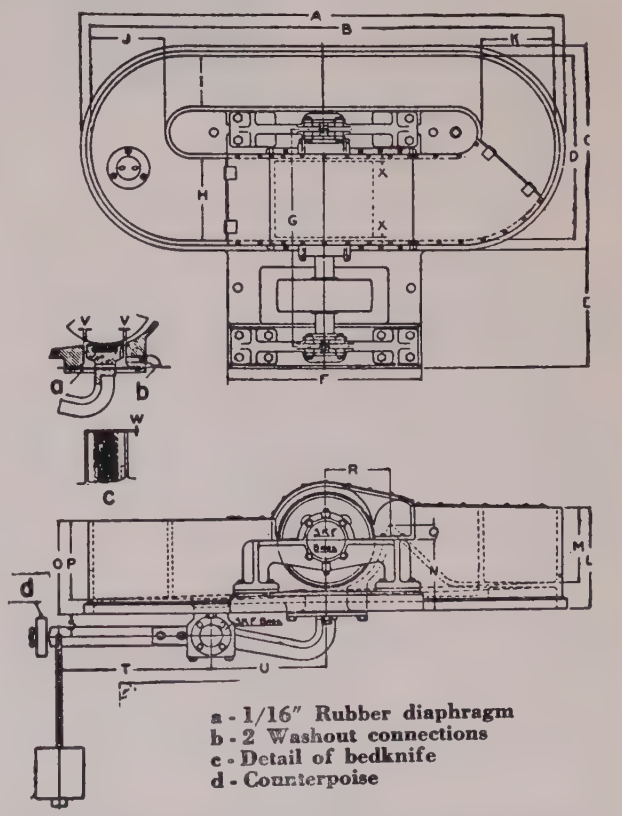
Presented at the meeting of the Gulf Coast Section of TAPPI held in New Orleans, La., May 15, 1959.

The Comparison of a Mead Refiner with a Valley Beater in Laboratory Evaluation of Pulp Strength Qualities

W. Earl Wright

THERE are three methods described in TAPPI Standard Testing Methods for the processing of pulp. These three methods are the beater method (T 200), ball-mill method (T 224), and the kollergang method (T 225). The two latter methods are suggested methods and the first method is an official standard method.

W. EARL WRIGHT, Gulf States Paper Corp., Tuscaloosa, Ala.



IRON TUB	TABLE OF DIMENSIONS OF NIAGARA LAB. BEATER												
SIZE · ROLL	A	B	C	D	E	F	G	H	I	J	K	L	M
1½LB. 7⅛" x 6"	37½	36"	16"	14½"	9¼"	15"	17"	6½"	4"	8¾"	5¾"	8¼"	6⅞"

H.P.	PULLEY	R.P.M.	N	O	P	Q	R	S	T	U	V	W	X
½	7⅛" x 2½"	500	5⅜"	7 5/16"	6 1/16"	1⅛"	5 1/16"	1¼"	17"	9"	3/16"	⅛"	¼"

Fig. 1. Niagara type 1½-pound beater with controlled bedplate

There are two methods suggested by TAPPI for routine control methods. RC 117 suggests the use of a Mead Corp. refiner and RC 118 suggests the use of a Morden laboratory refiner. It is the purpose of this paper to compare the test results obtained between a Valley beater and a Mead refiner and to offer suggestions for using the Mead refiner in routine control testing.

GENERAL DESCRIPTION OF THE PROCESSES

Figure 1 shows a view and dimenions of the Valley beater as described in TAPPI. The beater requires 360 g. of dry pulp at 1.57% consistency. An upward force of 10,700 g. is exerted on the roll of the beater by applying a weight of 5500 g. on the bedplate lever. The time required to obtain a beater curve on most kraft pulps, both bleached and unbleached, is approximately 40 min. for refining. Samples are taken at 10-min. intervals.

Figure 2 shows a view of the Mead refiner at Gulf States Paper Corp. The refiner is a disk-type refiner, where one set of slots set in a disk remains stationary while the other disk, containing slots, revolves.

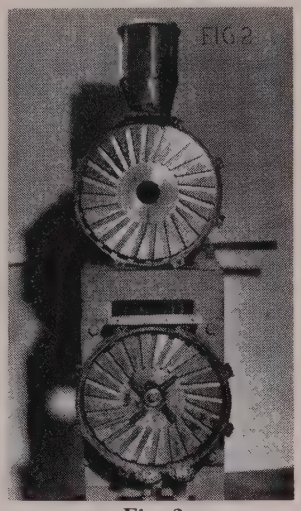


Fig. 2

A force is exerted on the stationary disk by applying a force of 5 lb. on an 18-in. lever arm against the rotating disk. Eighty grams of dry pulp, at a consistency of 2%, is required for each refiner load. Kraft pulp requires approximately 8 min. for refining to maximum burst and 300 to 200 CS freeness range freeness, when done in one operation and about 30 min. refining time in the refiner, when done at 2-min. intervals.

TESTING PROCEDURES

A standard pulp was used to calibrate the $1^1/_2$-lb. Valley beater as described in TAPPI Standard T 200 m-45. The procedure, forming handsheets for physical tests of pulp, T 205 m-58, was used for testing. Five handsheets were made at each 10-min. interval, and averaged. The weight was corrected to a 1.2-g. sheet.

TAPPI RC test 117 was used for the Mead refiner. Eighty grams of pulp refined at 2% consistency as stated. Intervals of 2 min. were necessary to establish a corresponding curve from the same pulp.

The curves shown are averages. The standard deviations are as follows:

Time, min.	*Burst, p.s.i.*	*Tear, g.m.s.*	*Freeness, ml.*
	Valley Beater		
10	3.1	16.6	10.3
20	2.6	8.9	24.0
30	2.4	8.1	35.1
40	2.9	5.2	27.9
	Mead Refiner		
2	5.2	20.0	11.6
4	3.1	9.0	20.0
6	2.5	10.2	57.0
8	2.2	8.1	33.0

The standard deviations for the Mead refiner were calculated from standard deviation formulas. More samples would probably lower the deviation some. It is noted that

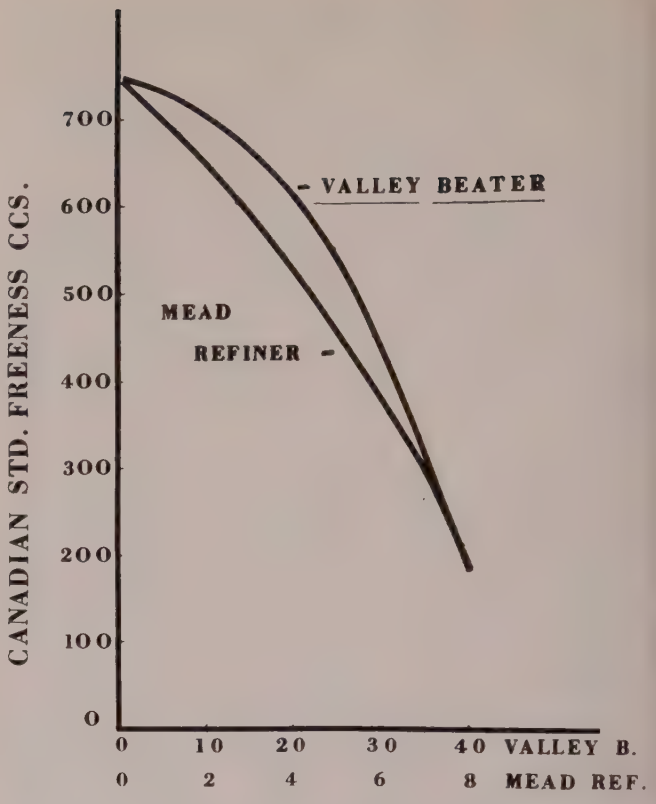


Fig. 3

the dispersion decreased as refining time increases and freeness decreases.

COMPARISON OF RESULTS

Freeness versus Time

When pulp is refined the freeness of the pulp, as measured by a Canadian freeness tester, decreases as work is done on the pulp per unit time. When about 200 freeness is attained the decrease in freeness per unit time is less. A comparison of the freeness curves of the Mead refiner and the Valley beater (Fig. 3) indicates 300 freeness is reached by the Mead refiner in one fifth the time it takes the Valley beater. Also, the Mead refiner and the Valley beater are refining together, i.e., the curves meet, at 300 freeness. At the beginning of refining, and until 300 freeness is reached, the shape of the

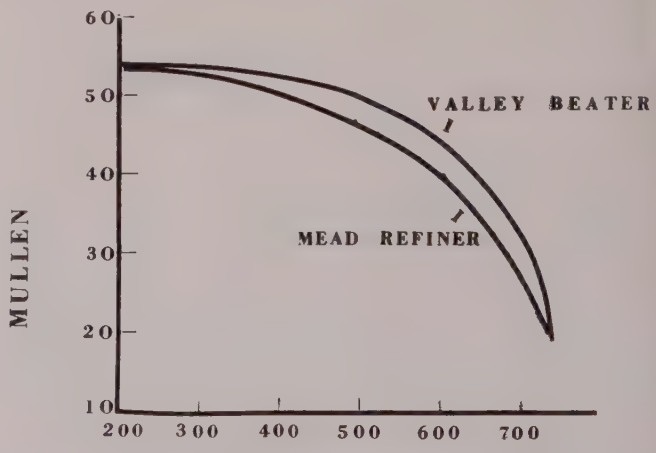


Fig. 4

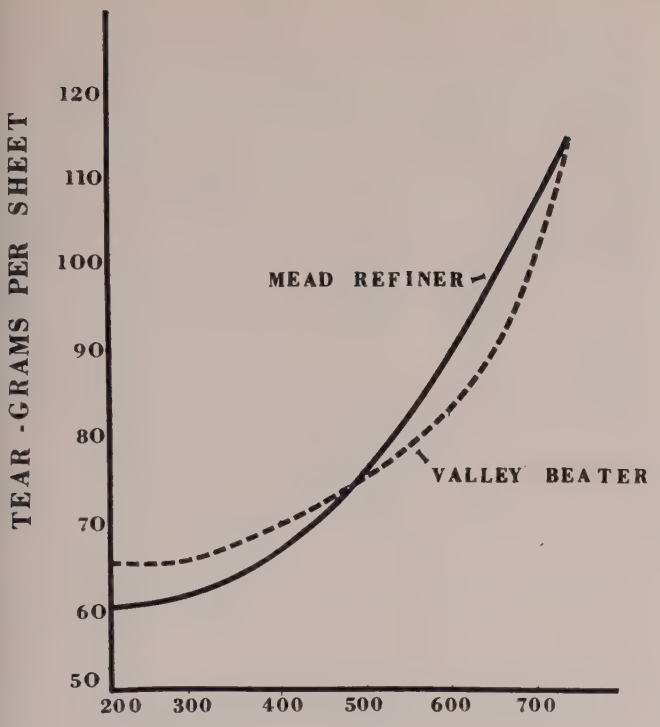


Fig. 5

curves indicate the Mead refiner is slowing the stock faster than the Valley beater.

Burst versus Freeness

As work is done on the pulp per unit time, as is shown on a beater curve, the bursting strength increases and reaches a maximum around 300 freeness and begins to level off between 300 and 200 freeness. A relationship between freeness and burst is shown in Fig. 4.

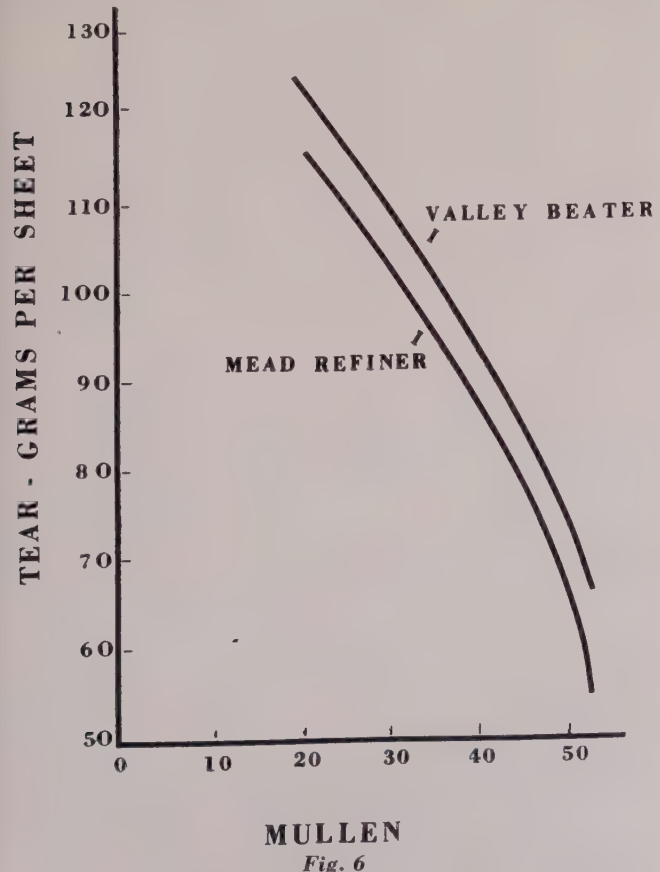


Fig. 6

A comparison of the curves developed by a Mead refiner and a Valley beater also indicates the bursting strength agrees most closely at 300 freeness or lower.

Freeness versus Tear

In developing a beater curve, the tear decreases as the freeness decreases and decreases as the burst increases. Figure 5 is a comparison of the freeness versus tear of a Mead refiner and a Valley beater. There is very little difference between the two curves. It will be noticed that the two curves cross at about 500 freeness and at no place on the curve does the tear differ by more than 5 points. The curves indicate, as stated before, that the Mead refiner is refining slightly faster at a given freeness than the Valley beater.

Burst versus Tear

The burst versus tear curves are similar in shape as is seen in Fig. 6. Here again the Mead refiner curve lies slightly under the Valley beater curve. However, their difference is very small, indicating there is very little or no degradation of pulp when refining with a disk refiner as compared to a beater-type refiner.

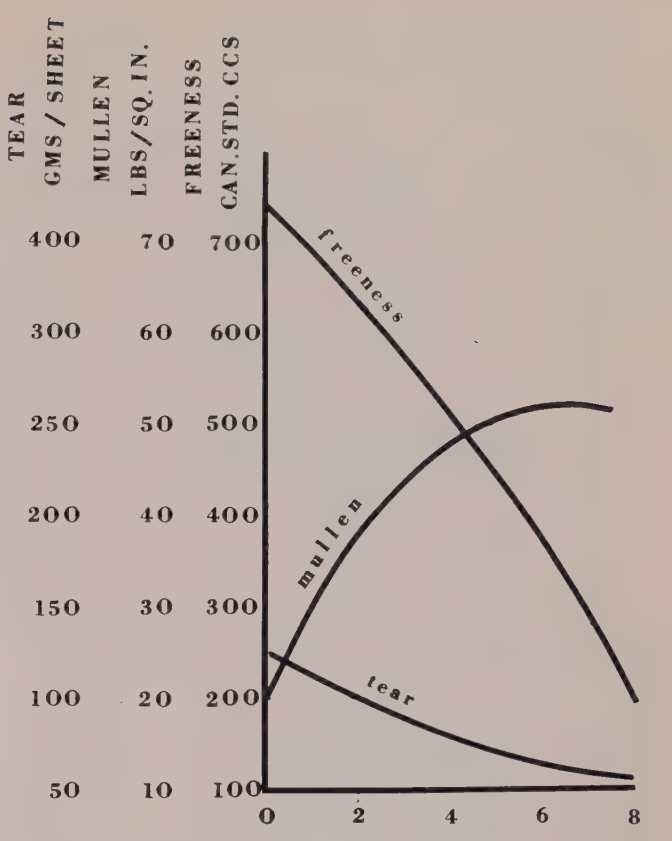


Fig. 7

CONCLUSIONS

1. A disk refiner of the Mead type requires about one fifth the time to refine a given pulp as compared to the Valley beater.
2. It is possible to substitute a Mead refiner curve for a Valley beater curve and vice versa with only a small error in the range of 600 to 400 freeness.
3. A comparison of the averages of a Mead refiner to a Valley beater indicates their difference in tests are least

between 300 and 200 freeness, and the deviation from average is less at this freeness.

4. It is practical to use a Mead refiner for routine control by refining to a predetermined point and comparing tests because: (a) the two types of refiner tests are similar, (b) time saved in determination is about one fifth, and (c) the refiner is self sharpening and does not have to be calibrated or checked as frequently.

Suggested Methods for Using the Mead Refiner in Routine Control

Referring to Figs. 3, 4, and 5 it is seen that for this particular pulp the burst and tear reach a maximum and minimum respectively at about 300 freeness and the Mead refiner tests coincide with the Valley beater tests at this freeness. It is also seen in Fig. 3 that the time necessary for refining to approximately 300 is 7 min. in the Mead refiner as compared to 35 min. in a Valley beater.

Using these advantages, it is suggested to refine to a freeness of 300 to 200 ml. and extend the curve in the general direction that is characteristic to that pulp until 300 freeness is reached, likewise extend the burst and tear curves in the same manner until they correspond to a 300 freeness and read off the respective tests (burst and tear) at 300 freeness. The small error in extending the curve would be insignificant as compared to errors in testing.

Since pulp strengths vary widely due to various characteristics such as bleach versus unbleach, hard cooks versus soft cooks, etc., it is suggested that a typical curve characteristic to a particular pulp be made as shown in Fig. 7. Obtain enough test results to give a reliable average and standard deviation. The limits could be set at 3 sigma or for closer control 2 sigmas or even sigma whichever is deemed sufficient in the 350 to 150 freeness range. Using this standard curve, make one beater determination, (in case shown in 7 min. refining) make five, handsheets from the pulp refined, average, correct to 1.2-g. handsheet and test in the usual mannner. Correct tests to 300 freeness and report as such.

Another suggested method of using a Mead refiner in routine control is in a sense a go-no go, or rather, a better than or worse than, a given standard.

The method of obtaining the standard curve would be the same as stated in the previous method. The number of minutes required to obtain 300 ml. freeness is used as a gage to determine the relative strength of a given pulp. Referring to Fig. 7 the required minutes necessary to reach 300 freeness is 7 min. A given pulp sample is refined for 7 min., five sheets made from the sample, averaged, and tested as before. A comparison of the tests after 7 min. refining is made by freeness versus burst and freeness versus tear as shown in Fig. 4 and 5.

Presented at the metting of the Gulf Coast Section of TAPPI held in New Orleans, La., May 15, 1959.

Comparison of Wet Pad Colormaster Brightness with Dry Pad G. E. Brightness

F. D. Hall

The Colormaster is a tristimulus filter colorimeter,* designed primarily to measure small color differences between materials of elosely similar colors. It can also be used as a brightness tester by taking the blue filter reading as the brightness.

Since wet pad Colormaster brightness tests are used for routine control in our bleach plant and G.E. brightness tests are made on reels from the paper machine, it was necessary to establish a relationship between wet pad Colormaster brightness and dry pad G.E. brightness.

F. D. Hall, Project Coordinator, Container Corp. of America, Brewton, Ala.

* Made by Manufacturer's Engineering and Equipment Co., Hatboro, Pa.

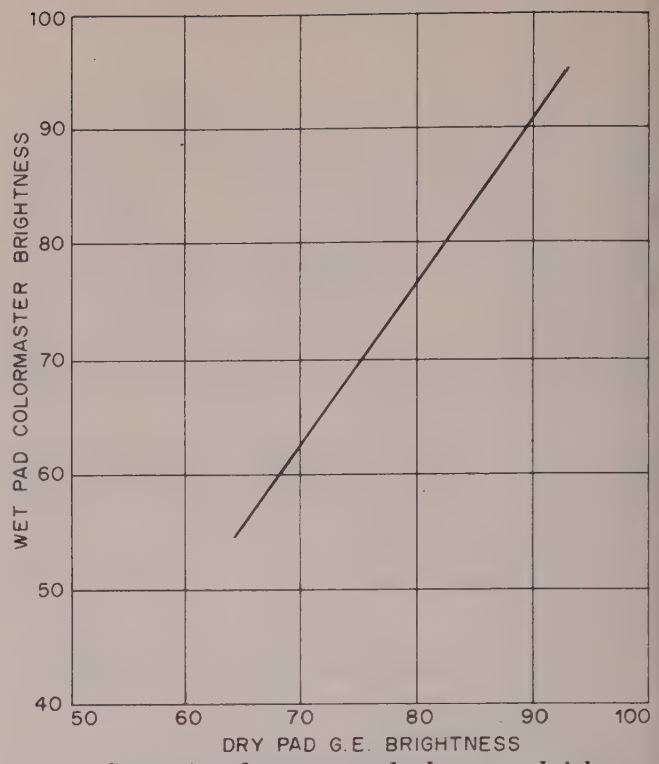


Fig. 1. Conversion chart—wet pad colormaster brightness to dry pad G.E. brightness

As part of the correlation work, a study was made of several variables in the procedure for determining wet pad Colormaster brightness. It was concluded that the major source of error is the readings taken on the pad itself, probably due to variations in the pad surface. Variations in procedure appeared to have little effect on final brightness.

The relation between wet pad Colormaster brightness and dry pad G.E. brightness, both determined according to the attached procedure, was found to be good.

EFFECT OF VARIATIONS IN PROCEDURE

The following variables were studied in the procedure for wet pad Colormaster brightness:

1. *Color of backing materials.* A white block behind the wet brightness pad gave an average increase of 0.3 to 0.4 point brightness compared to a gray block.
2. *Weight of pulp used.* A 60-g. hand-squeezed sample gave a brightness 0.2 point higher than a 40-g. hand-squeezed sample.
3. *Weight applied to pad.* Use of a 3000-g. weight gave a brightness 0.2 point higher than did the use of a 1000-g. weight.
4. *Amount of sample filtrate used in forming pad.* No difference in brightness between 500 and 1000 ml. of filtrate; pad formation is more difficult with the smaller quantity.
5. *Number of blotters used.* Use of one blotter on each side, and two passes through the wringer (changing blotters after the first pass) gave a brightness 0.3 point higher than using two blotters on each side and one pass.
6. *Precision of the method.* Seven pads, made from the same pulp sample, were all within 0.7 point in brightness.
7. *Readings on a single pad.* The average range of five readings on one pad (21 pads tested) was 1.3 points; maximum range for one pad was 2.5 point. For dry G.E. pads, the average range of four readings on one (21 pads tested) was 1.6 points; maximum range for one pad was 3.8 points, minimum range was 0.5 point.

The above tests were made using one pulp sample from the last stage in the bleach plant. The data indicate that variations in procedure are less important than the variations

in the readings on a single pad. These latter variations are probably due to a nonuniform pad surface; as the pad dries, these nonuniformities become more pronounced, causing a wider variation in the dry pad G.E. readings than the wet pad Colormaster readings. In addition, the larger sample opening in the Colormaster probably has an averaging effect.

Since seven pads from the same pulp sample gave brightness values within 0.7 points, it was felt that the wet pad Colormaster method is sufficiently precise for control purposes. This applies, of course, only when the method is accurately followed. The procedure for dry pads for G.E. brightness is very similar to the TAPPI Standard Method.

CONVERSION CURVE: WET PAD COLORMASTER BRIGHTNESS TO DRY PAD G. E. BRIGHTNESS

The conversion curve included in this paper was made using the attached pad making procedure. The curve is valid only for the Colormaster instrument under consideration.

Nineteen pairs of wet and dry pads were used to form this curve; the range covered was 57 to 91 points on the Colormaster. The curve was drawn using single regression statistical analysis. The correlation coefficient was 0.9888; a coefficient of 1.000 is considered perfect correlation.

PROCEDURE FOR MAKING BRIGHTNESS PADS FOR COLORMASTER AND G.E. COMPARISON

A. Procedure for Colormaster (Wet Pad)

1. Collect at least a 2-l. sample of slush pulp.
2. Form wet pads for Colormaster reading.

a. Weigh out 50 g. of hand-squeezed pulp.
b. Add 1 l. of filtrate collected from sample.
c. Agitate thoroughly in beaker with stirring rod.
d. Place one sheet of 15-cm. Whatman no. 4 filter paper in Buchner funnel, wet paper with sample filtrate, turn on vacuum, and pour in pulp slurry.
e. Let water drain from pad until air pulls through pad and water comes out in drops.
f. Invert funnel over a sheet of 18-cm. Whatman filter paper and shake pad onto the filter paper.
g. Place two blotters on each side of pad (still covered with filter paper) and put through the wringer once.
h. Remove blotters and 18-cm. filter paper, keeping fingers off the surface to be tested.
i. Standardize the Colormaster.
j. Place wet pad on Colormaster, with the 15-cm filter paper on top (do not fold the pad). Place the gray standard on top of standard.
k. Read brightness at 5 points on the pad and average results.

B. Procedure for G.E. (Dry Pad)

a. Weigh out 25 g. of hand-squeezed pulp.
b. Add 700 ml. of filtrate collected from sample.
c. Follow steps 2c through 2g in above method.
d. Remove blotters and place pad in drying rings so that it is supported by the 18-cm. filter paper.
e. Let pads stand until dry.
f. Remove pads from rings, remove both filter papers and cut into $1^1/_2 \times 2$-in. squares. Use the squares for readings backed up by the edges.
g. Read G.E. brightness on the four square tabs, and take average.

Presented at the meeting of the Gulf Coast Section held in New Orleans, La. May 15, 1959.

Fourth Deinking Conference

Roger Smith Hotel, Holyoke, Mass.
Oct. 8–9, 1959

Aluminum Foil Standardization of Jumbo Mullen Tester

J. B. Rhyne

THE use of the Jumbo Mullen tester for testing the bursting strength of linerboard is a very common and important practice today. It is almost universally accepted as the standard test. But there is quite a bit that is still not standard about the test. One of the main problems that we have encountered is how to determine when the Mullen tester deviates from the normal. Since there are so many possible variables that can get out of adjustment, we have set two specifications on each tester.

Specification No. 1

The extension of the diaphragm to a height of 1.8 cm. should produce between 40 and 45 p.s.i. on the gage. Although this is a very important step in the maintaining of a Mullen tester, I will only mention it to point out that the tester must first meet this specification before it is checked for Specification No. 2.

Specification No. 2

Aluminum foil has been selected instead of paper because it is not affected by a variation in humidity. A 3-mil. aluminum foil strip 4 by 24 in. is popped eight times. The average points of Mullen must be within 3 points of the previously arrived at number. This number is derived from a bundle factor multiplied by the weight of the individual strip. The factor is derived by selecting 20 strips from a new bundle of approximately 450 strips at nearly equal spacings through the bundle. The position of each sample strip in the bundle is marked with a numbered tag and the corresponding number is marked on the strip. The strip is then calipered. After rolling the strip on a $^1/_2$-in. diam. for ease of handling, they are weighed and popped on a Mullen tester, which we call our standard tester. This tester is used only for standardizing the aluminum foil strips and a very few other special tests. The diaphragm and gage are checked before use. The factor is the average Mullen of the 20 strips divided by the average weight. If any of the strips do not conform with the other test strips, the bundle is regrouped by segments, as marked by the numbered tags, into similar categories. The random selection and factoring is then repeated for each category.

This method allows us to iron out many of the discrepancies due to caliper and weight disagreements in a bundle. By setting certain specifications for the aluminum foil such as being polished on both sides and by watching the caliper very closely, we have been able to get a foil that will give good checks. We try to maintain a foil that will give an average Mullen of 100. However, uniformity is more important than the range; and most of our work has been along these lines.

J. B. RHYNE, International Paper Co., Panama City, Fla.

Presented at the meeting of the Gulf Coast Section of TAPPI held in New Orleans, May 15, 1959.

Use of the I.G.T. Print Tester

Thomas E. Linden

THE prediction of printing results on paper is extremely difficult. Until recently a number of properties affecting the printing quality were measured separately such as strength, hardness, and smoothness. Unfortunately, a given apparatus does not always measure the properties for which it was designed. It is not surprising that considerable differences between the predicted and the actual results were often found. This method therefore is both unreliable and tedious.

THOMAS E. LINDEN, Assistant Chief Chemist, International Paper Co., Moss Point, Miss.

There has been a definite need for instruments by which various factors may be determined in advance of actually having the printer apply the ink to the paper and noting the results.

An instrument has been developed by the Institute for the Graphic and Allied Industries in Amsterdam, Holland, known as the I.G.T. tester which can be used to measure various qualities of paper that are to be printed. At our plant at Moss Point we use the I.G.T. print tester primarily to measure the picking resistance of paper—and I would like to confine most of my remarks along this particular line.

The I.G.T. print tester consists of two parts; an inking unit and a printing apparatus. The inking unit consists of two inking systems each of which comprise two steel rollers and a composition roller. An accurately measured quantity of ink is evenly distributed over the composition roller with which the printing form, a brass disk, is then inked during a certain space of time. The layer of ink on the disk is in this manner kept constant.

The printing disk is then placed in the printing apparatus which provides the counterpart of an impression cylinder in the shape of a sector of 150°. The sector is covered with a manila or offset blanket. On the other side of the apparatus, the spindle to which the sector is attached carries a weighted pendulum. When released the pendulum descends with increasing speed. The pressure exerted on the offset blanket by the printing disk can be adjusted by means of a hand knob on the tester.

The strip of paper to be tested is placed on the blanket and secured with a clamp at one end of the sector. The inked disk is in touch with the bottom of the strip. The print is produced by releasing the pendulum. The initial printing speed is nil. It increases with the descent of the pendulum and reaches its maximum when the pendulum has reached its lowest positon.

The greater the printing speed, the sooner picking occurs. Highly pick resistant paper, therefore, only pick at high printing speeds. Consequently, the distance between the bottom of the test strip and the point where picking is observed express a certain degree of pick resistance. Using the velocity versus segment displacement curves furnished with the instrument we express the pick resistance in terms of printing velocity (feet per minute) for a given tack ink.

In our initial laboratory evaluation of this instrument at Moss Point standard inks bearing tack numbers from 1 through 7, made by the Printing Ink Div. of Interchemical Corp. were obtained for use with the tester. In order to obtain satisfactory test results on coated paper it is sometimes necessary to use a higher tack ink on one group of samples and then a lower tack ink on another group. If the results were reported as actual picking velocity for the ink being used, there would be difficulty in comparing the quality of the two samples which had been tested with different inks. For this reason, the picking velocities obtained on the same paper with different tack inks were compared to see if a constant relationship existed between the inks. With this in mind we were able to express our data mathematically into conversion tables so that results obtained with any of the inks can be reported on the tack no. 4 ink basis.

Prior to using the I.G.T. print tester at the Moss Point mill our basic test for measuring picking resistance of a coated paper surface was the Dennison wax pick. In our initial study of the I.G.T. tester experimental work was conducted to determine the relationship between picking velocity and Dennison wax pick numbers.

Samples from approximately 30 different runs of paper were tested for I.G.T. picking velocity and Dennison wax pick. Graphs of our test data show that there is good correlation between the two for our casein adhesive coatings. The Dennison wax test is not thought to be reliable for use in testing coatings containing latex-type adhesives.

Further experience in testing samples from each reel in a run, however, shows that occasionally the I.G.T. test will show a slight, but definite, change for a reel that may not be accompanied by a change in the Dennison wax number. It is felt that the I.G.T. picking velocity results, when obtained on Moss Point coated grades, are probably more precise than the Dennison wax pick results.

A convenient attachment to the I.G.T. tester is the spring drive device. The speed limit of the standard printability tester is about 1.1 m. per sec. This speed does not match the behavior of paper and ink at high speeds on modern rotary presses. With this in mind a spring drive device has been designed by means of which the printing speed can be pressed up to 3.2 m. per sec.

Our experience with the I.G.T. print tester at Moss Point has been gained by using this instrument primarily to measure picking resistance. Available literature indicates the apparatus can be used for other purposes in evaluating paper quality such as: (1) investigation of the density of the print, (2) the striking-in of ink during the first minute, (3) determination of the drying time of inks, and (4) evaluation of the smoothness of paper.

The I.G.T. print tester has several important advantages as a test instrument. It is compact. Printing pressure, thickness of the ink layer, and printing speeds can be kept constant or quickly changed with practically no difficulty. Only a very small amount of ink and paper are required. The tests only take a short time.

The use of this instrument at our mill leads us to the conclusion that the I.G.T. print tester can be a reliable means of measuring picking resistance of coated papers. With its numerous advantages, the I.G.T. print tester represents an instrument with which the paper industry should be able to predict printing results with some degree of certainty.

Presented at the meeting of the Gulf Coast Section of TAPPI held in New Orleans, La., May 15, 1959.

Betameter Chart and Plastic Ruler Method as an Aid to Basis Weight Control

T. M. Cunningham

A FEW months ago we were surprised to receive a complaint on excessive basis weight variation from a particular customer, because our tests had shown that every reel run into the order was up to specifications. However, to maintain customer satisfaction and to obtain information which might explain the difference in basis weight, our quality control supervisor visited the customer's plant.

Our supervisor reported that the difference in testing methods would probably explain the excessive basis weight variation. The customer's method consisted of weighing a single ply sample of approximately (10) sq. in. taken at close intervals across the rolls. At the mill, sufficient weight sheets of 2 by 3 ft. area are torn from the reel so as to give tests across the entire reel. The average value of these individual basis weights is posted as the reel basis weight. Recheck tests were made on our file samples which confirmed the customer's and the mill's test results. Hence, it was apparent that there were light and heavy areas in the sheet which were out of specification, but these areas were sufficiently distributed so that on mill size samples the tests were up to specifications.

Our problem was to devise a test to measure either the basis weight or variation in basis weight at frequent intervals across the reel. Either type of test would give data which could be used to improve the sheet weight variation. Since our paper would be subject to test at 2-in. intervals, we made a small tear-out template (2 by 4.762 in.) and made consecutive basis weight tests across the entire reel. After testing

T. M. CUNNINGHAM, International Paper Co., Mobile, Ala.

two reels we were convinced that this method was impractical for our use and another method was necessary.

For several years now we have been using an instrument which makes a profile chart that indicates variations in the mass content of a strip sample when it is fed through the instrument. This instrument, the Foxboro beta ray sheet profiler, operates on the absorption properties of paper. The measuring unit is composed of a radioactive source eminating beta rays and an ionization chamber as a detector. As the mass of an absorbing material between the source and the receiver increases or decreases, a strip chart graduated in 1% intervals records these variations. The profiler's ability to accurately indicate basis weight variations was checked by making a profile of a reel strip and then making individual moisture-free basis weight determinations every 2 in. across the strip. The results of this check were good.

This gave us a rapid dependable method of obtaining basis weight variations, but it was necessary to provide a convenient method of converting these data into usable information for the machine crew. This portion of the problem was resolved by making a special width transparent plastic ruler to be used when analyzing the reel profiles. The width of this ruler was determined from the profile chart and the basis weight limits for the particular basis weight paper being tested. If, for example, the basis weight limit specifications were ±4% of the average basis weight, then the ruler would be made eight percentage units wide as measured from the profile chart.

In analyzing the profiles, the imaginary center line of the ruler was placed so as to coincide with the sight line average of the curve. Any portion of the curve that extended beyond the edge of the ruler indicated an out of specification area, and proper adjustments should be made on the machine. The actual position on the machine which needed adjusting was readily pinpointed by the chart-strip feed relationship in which each $^1/_{10}$ in. chart travel was equivalent to 1 in. of strip travel.

By the use of profile chart and plastic ruler, paper machine adjustments were made which resulted in decreased sheet variation thereby solving our problem.

Presented at the meeting of the Gulf Coast Section of TAPPI, held in New Orleans, La., May 15, 1959.

A Device for Measuring the Curling Tendency of Paperboard

L. L. Clapp

To ALWAYS produce a sheet of paperboard which will be perfectly flat is the intent of almost all manufacturers of this product. This degree of perfection is not always attained. To determine how far the product departs from the ideal flat condition is no great problem in surveying. To measure this deviation several devices may be employed having high degrees of precision but which also are either expensive to purchase or tedious to operate. In order to save money and to provide an instrument rugged enough for control testing and at the same time simple in operation, a device was designed for measuring this property. The prototype was constructed in our machine shop at Pensacola. No novel or unusual methods of mensuration are involved. The only claim made for it is that if a circular specimen of paperboard of a given size is laid on it with the concave part of any curvature upward, a fairly precise measurement of how far the edges stick up can be made without much effort.

The device has four principal parts: a turntable mounted on a spindle free to turn in a sleeve, a microscope with a cross-line reticle, a micrometer screw, and a support for these components. The microscope is mounted at the side of the turntable so that its axis is parallel to the surface of the turntable and intersects the axis of the spindle. The micrometer screw is arranged below and in contact with a ball bearing inserted in the lower end of the spindle and is used to raise and lower the turntable and measure its elevation.

We use the instrument in this manner: a paperboard specimen 8 in. diam. is laid centrally with its concave side upward upon the turntable, the microscope is focused on the upper edge of the sheet, and the turntable is rotated until a low point on the edge is located. The micrometer screw is used to raise or lower the turntable and specimen until the edge is aligned with the cross-line of the reticle. With a bit of practice the location of the low point and the alignment of the cross-line can be done simultaneously. The micrometer barrel is read and the elevation recorded. The turntable is rotated until the opposite low point is reached and its elevation is likewise determined. The same is done for the high points. Half the difference between the sums of the low point readings and of the high point readings is used to express the amount of curl or warp of the specimen. Which ever side shows the concavity is also noted.

Several modifications in equipment and procedure have been suggested. One which we plan to make is that a circular scale of 360° or of 12 points be attached to the edge of the truntable and the sample be oriented so that its machine direction will coincide with the zero axis of the scale. In this way the highs and lows can be reported together with their locations so that the direction (either machine, cross, or diagonal) of the curvature can be expressed. This procedure will allow also that the presence, location, and extent of more than two highs and two lows can be measured and expressed.

In conclusion, we feel that with this simple and inexpensive device measurements of warpage or curl can be performed conveniently and with fair precision. Whether the measurements are to be made on specimens which have been exposed to standard testing conditions until equilibrium has been achieved, at various levels of temperature and relative humidity, or at intervals of time after manufacture is felt to be outside the scope of this discussion and would best be left to the discretion of the persons intending to use it.

Presented at the meeting of the Gulf Coast Section of TAPPI held in New Orleans, La., May 15, 1959.

The Use of Drainage Test with High Yield Kraft

Leo Shealy

BEFORE the advent of high yield kraft, very little, if any attention was given to drainage rates or other physical variables that would be characteristics of stock from the digesters. These variables which still exist under most mill conditions can only be partially controlled at the digesters and further back in the system. With high yield pulp, less chemical reaction that has taken place in the digesters, thereby, a reduction in per cent rejects or increase fiberizing must come from some mechanical action on the pulp after digestion. The need for some method of control of refining became apparent when we started cooking high yield pulp.

It was apparent that a freeness test or drainage test should be used for this control. It must be stated at this point that most control tests in the paper mill are used as past record. They are mostly used to see what has already happened so that adjustments can be made and records kept to keep variables within reasonable limits.

With the above reasoning in mind, a drainage test and tester was designed to be used for this purpose. It was decided the test should be simple and reliable. After a number of trials and errors using various sizes and shapes of testers, a tester was found that would be satisfactory and reliable. The tester was made of 2-in. stainless steel pipe

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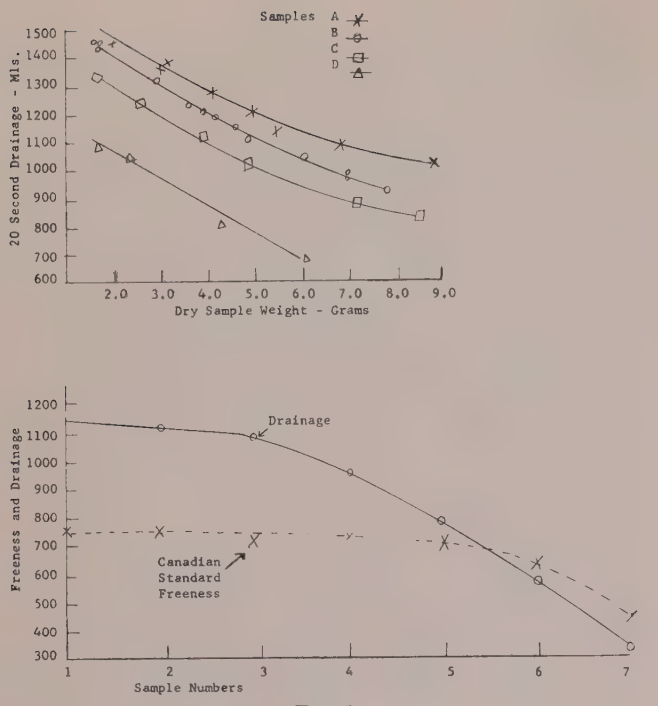


Fig. 1

with capacity of 1600 ml. There is a removable screen on one end and removable cap with petcock on the other. The tester is mounted so that it pivots in the middle so that it can be vertical with either end down.

The procedure for running test is to fill with diluted stock with petcock closed and in bottom position. Place screen on tester and invert. Open the petcock and record milliliters drained in 20 sec. The pad is then removed from the tester, dried, and weighed. From the nomograph a corrected drainage to 4.0 g. is used for the final result.

We have found very good correlation with sheet weights between 2.0 and 5.0 g. converted to a 4.0-g. basis. As can

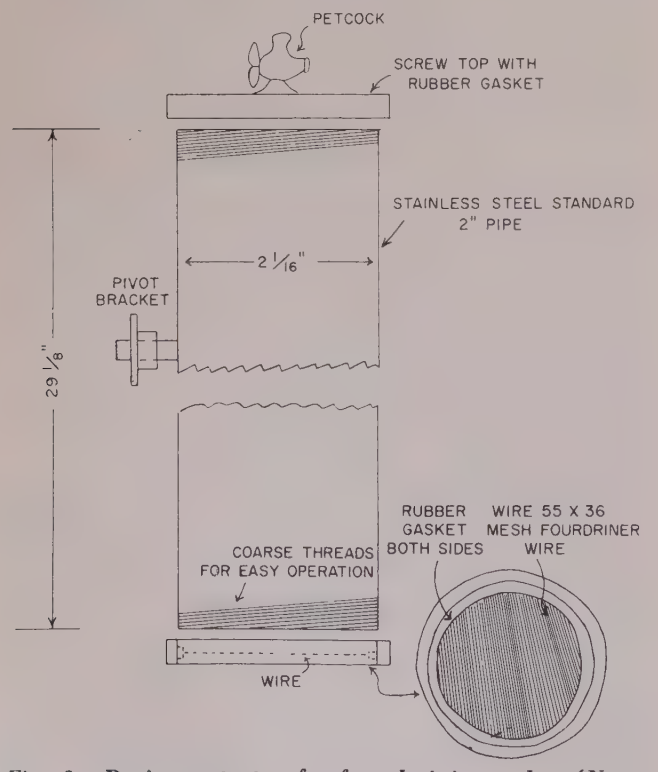
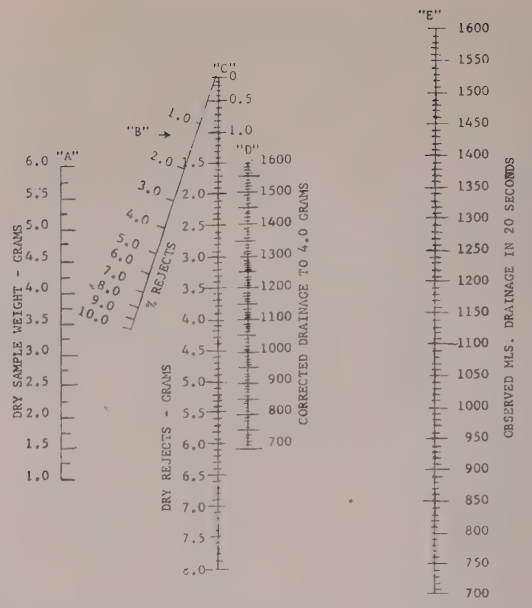


Fig. 2. Drainage tester for fast draining pulp. (Note: Tester with bottom cap and seal cap in place should hold 1600 ml.)



PROCEDURE:

Weigh 400 grams stock from 3rd stage. Mix and remove 1 liter after diluting to 11 liters.
Screen remaining pulp slurry. Dry and weigh rejects._____"C"
Dilute 1 liter portion to 2 liters. Use 1600 mls for drainage test.
Record observed 20 seconds mls. drainage. _____"E"
Dry pad from drainage test._____"A"
Draw straight line from "A" to "E". Read correct drainage at "D".
Draw straight line from "A" to "C". Read % rejects at "B".

Fig. 3

be seen from Fig. 1, the slope of the curves is very close to the same value. The slope used for plotting the nomograph and arriving at a formula was taken as an average of the curves between 3.0 and 5.0 g. The slopes were as follows: curves A = 0.47; B = 0.50; C = 0.47; D = 0.56. The average slope being 0.50 or slope of curve B. Since this curve was the middle range, we were interested in the values taken from this curve for calculations to follow.

The formula for curve B of Fig. 1 would be as follows:

Let Y = corrected drainage to 4.0 g.
Y_1 = observed drainage
X = Corrected pad weight 4.0 gr. (ovendry)
X_1 = Sample weight in grams (ovendry)

From curve B then

at 4.0 g.-drainage = 1200
at 3.0 g.-drainage = 1300

It follows that:

$$Y - Y_1 = M(X - X_1)$$
$$M = -100$$

Substituting back the formula would be $Y = Y_1 + 100 \times 1 - 400$

The nomograph was plotted from the above information and methods from "Empirical Equations and Nomography" by Dale S. Davis

The drainage test was found very helpful for controlling the refining in the wash room prior to washing. From the data compiled on drainage, per cent rejects, and washer performance, it was found that it was possible to lower drainage rate at a much faster rate than per cent rejects were lowered after a certain point was reached in refining. This would cause low production rate on washers and considerable variation in refining at the paper machines.

Another line was then added to the nomograph to include per cent rejects. The procedures for running these tests are shown at the bottoms of the nomograph.

As we all know, the Canadian Standard freeness tester was designed to be used with much slower stocks than will be found in the raw stock from high yield refining. To show the characteristics of the drainage tester, seven samples were collected at various places in the system which give the relationship between Canadian Standard and St. Joe drainage

tester. All tests were made with 4.0 g. on drainage tester since low drainage characteristics of the tester have not been explored; therefore, no correction was necessary.

We hope these data and procedures will be helpful to anyone with this type of problem. The drainage test has proved satisfactory for controlling wash room refining and having data available for trouble shooting problems that do arise. It is through the "use" of proper control testing that a quality product is made.

Presented at the meeting of the Gulf Coast Section of TAPPI held in New Orleans, La., May 15, 1959.

Recent Developments in Paper Coating Additives

Clark E. Snook and R. B. Porter

As you might guess from the title of this paper, our particular experience in the coating field has been in the application of additives to coating formulations. What is a paper coating additive? To us a paper coating additive is a material or mixture of materials that is added to the color, generally in quantities less than 5% based on the weight of the pigment, for the purpose of improving not only the physical properties of the coating but also the operation of the coating process. These materials or mixtures are either water soluble or water dispersible. Some of the properties of a coating modified by the use of additives include: (1) water resistance, (2) viscosity, (3) plastic flow, (4) dusting, (5) levelness, (6) wet rub, (7) foaming, (8) color, and (9) whiteness. It is because of the modification of some of these properties by the use of additives that improvement in the printability, feel, and appearance of the coating is obtained.

GROWTH OF COATED PAPER

The production of coated paper has grown at a rate considerably greater than the growth rate of the industry in general. Starting with the introduction of machine coating in 1930, by the "Massey" or "Consolidated" coater, the tonnage of coated paper has increased from about 200,000 tons up to approximately 3,000,000 tons per year. This phenomenal growth has been due to many factors not the least of which has been the tremendous technological advances in the coating field. Not only has there been the development of synthetics, such as the acrylics, butadiene-styrene, vinyls, and many others, but also the development of methods and equipment so that today there are available processes for applying almost any type of coating. There is no doubt that the developments to come to the coating industry during the next 20 years will surpass those of the past two decades.

Most of this increased production was in lightweight papers for magazine, book, etc. However, today more and more board is being coated. As one coating expert has said, "there is probably not one southern kraft mill that is not on the brink of coating or not experimenting with coating. I woundn't be surprised if some day coated board surpassed coated printing papers."

Of course, a little additional incentive to the high interest in the coated board field is the competitive picture, particularly in the packaging field. The plastics and aluminum foil industries are very active in this field due to the trend to more self-service stores where packaging appeal is an important sales feature.

EQUIPMENT FOR COATING

As in any industry higher production per machine will tend to reduce cost of the product, but increasing the production and, therefore, the speed of a machine results in coating application problems especially with high solids coatings (60%). Variations in the type of coating equipment, the rheology of the coatings, the properties of the base sheet, and other factors make the perfect coating difficult to obtain. Improvement in flow properties, flexibility, leveling, dusting, etc., are still to be desired. Successful coating is obtained by the application of a smooth and constant amount of color to a sheet of paper or board. Basically, all coating is concerned with three-unit operations: (1) metering, (2) application, and (3) smoothing.

Many types of coating equipment are available, such as the "size press" which is one of the oldest and most commonly used method of machine coating at low solids (40%). The most recently developed equipment is the "trailing blade" coater. The "trailing blade" has created considerable interest because of its versatility and the fact that metering, application, and smoothing of the coat are accomplished simultaneously. No doubt most of you are familiar with this coater and are aware of its principle of operation. Basically, the sheet is passed around a rubber covered roll, through a reservoir of the coating and under a flexible blade which serves as the bottom of the reservoir. The "trailing blade" coater has no apparent speed limitations, in fact, it has been indicated that the higher the speed the better the coating. Speed is governed largely by drying capacity. The range of coating solids that can be used with the coater varies from 0 to 65–70%. Although it was first thought the coater could apply only lightweight coatings (2 to 5 lb.), coating weight up to 10 lb. per side has been applied. Another feature of this coater is that many times calendering of the coatings can be minimized or even eliminated. As with other types of coaters, coating additives have proved helpful with the coating process.

THE ROLE OF COATING ADDITIVES

In spite of the best advances made to date in coating methods and formulations, the customer still desires that little extra or plus value which may be the hinge for a sale. We like to think of coating additives in this way because many times (not always) a coating can be upgraded by the use of the right additive.

Pattern Formation or Nip Pattern

It is almost impossible with the present coating methods to produce a coated sheet that does not show a typical pattern formation. This pattern is scarcely visible in the case of cast coated paper, but is much more pronounced in the case of roll coated paper. In any event, it is possible to trace the type of pattern to the method of coating used. By studying coated papers under ordinary light, black light, and microscope, the experts can identify the type of coating method.

From the printability standpoint patterns are very undesirable mostly because they result in a nonuniform sheet. Smoothness, ink receptivity, printing pressure, and other printing properties are all directly affected by the pattern. If the pattern is too pronounced, it may not even be possible to print the sheet successfully.

As we know, pattern formation is caused by many variables, such as the rheology or flow characteristics of the coating color. Patterning results from film splitting, that is, the way the film of coating leaves the nip and is laid down on the surface of the sheet. The smaller the breakaway angle, the more the nip pattern. Conversely, the larger the breakaway angle, the less the nip pattern.

In addition to film splitting, there is the problem with tension ridges. When the film of coating splits away from the nip onto a moving web of paper, the film breaks into fine ridges (tension ridges) parallel to the machine direction. The breakaway angle and degree of tension ridges can affect the pattern and consequently the printability. Surface discontinuities can also be caused by the presence of air bubbles or droplets of hydrophobic materials present in the coating. Casein dispersions and some of the synthetic latices, as you

Clark E. Snook, Technical Engineer, and R. B. Porter, Technical Manager, Paper Chemicals Dept., Nopco Chemical Co., Harrison, N. J.

probably know, have a very strong tendency to entrap air whenever they are agitated. Air in coatings cause small pinholes or craters when a bubble breaks near the surface. In the printing process ink may not reach the bottom of these craters, hence will show as white spots in printed copy.

From this brief discussion of some of the causes for a non-uniform coating surface, it is readily apparent that materials which can be added to the basic coating ingredients to eliminate or lessen these difficulties, can be most helpful in producing a smooth, uniform coating. Very effective defoamers and leveling agents have recently been developed to assist the coater in overcoming some of his problems with coating patterns. The use of these additives can result in that plus value that will please your customer.

Dusting in Supercalendering

The supercalendering operation exerts large stresses on the coating while smoothing its surface. It is not uncommon for fine particles of pigment to be broken away from the matrix of adhesive by these stresses causing the well-known dusting phenomenon. R. A. Diehm at Western Michigan College, Kalamazoo, Mich., attributes dusting to a condition known as flooding. Flooding is the separation of the pigment and adhesive during the drying operation and is probably related to the water retentive properties of the adhesive and water absorption of the base sheet. Since starch coatings dry more rapidly than casein coated sheets, dusting can be a greater problem with starch coatings. Flooding causes differences in ink absorption of the dried coating and may cause a serious dusting condition on the supercalender. When the coating is applied uniformly to a moving web, according to Diehm, some water is immediately absorbed into the sheet as well as some water being evaporated by drying. Water vapor comes up through the coating carrying with it some of the pigment fines. Thus the top of the sheet is covered with pigment without adhesive and serious dusting results.

When paper is printed by letterpress, the ink on the type face lifts this dust from the printed areas of the sheet. It works its way back through the ink system and causes trouble. Dust is even more troublesome in the offset process. Not only the inked areas, but also the moist uninked areas pick up dust from the paper. Since the offset process depends upon intimate contact between the sheet and the rubber blanket carrying the ink, a very small amount of dust on the blanket causes serious trouble.

Dusting can be such a problem in the supercalendering operation as to cause a cloud of fine particles to build up in the area and slow down production or even temporarily stop the calendering operation. However, the effect such dusting may have on printability is even more serious. Here a coating additive can be helpful. The use of metallic soaps such as calcium stearate has proved to be an effective method of decreasing or eliminating dusting. One theory proposes that the particles of metallic soap builds structures in the coating that streaming water has to go around so that the sheet is closed by drying before flooding can occur.

Calcium stearate is unique and can be used in either the dry form or in the recently developed fluid dispersions or emulsions. The particle size of these dispersions is in the range of the finest clays, 2 to 3 mu with a melting point of 150° C. It not only will help a dusting condition but has a bulking affect and adds to the resiliency of the coating which aids good reproduction in printing.

It also has antiblocking and lubricating properties that aids gloss in the supercalendering operation. Its effect as an antiblocking agent is particularly noticeable in latex-casein coatings for the bronzing process and cast coating.

Another feature of metallic soaps is their ability to assist the passage of clay particles through the nip that otherwise would be rejected. Very little soap, generally less than 2% on the weight of clay, is required. The effect of the soap is quite noticeable by the fact that very little settling of the larger clay particles is evident in the tub.

In general, about twice as much calcium stearate dispersion can be added to a coating compared to other additives such as soaps with less affect on viscosity.

Another common additive to coatings is soap, such as sodium or ammonium stearates. This material acts as a lubricant, viscosity modifier, and wetting agent. Many times improvement in the finish of the coating is obtained by the judicial use of these or similar soaps. Too much soap may cause a livering condition that is a buildup of a gellike structure throughout the color which results in a substantial change in viscosity.

A recent development in this type additive is a 30% solids fluid dispersion of ammonium stearate. In contrast to the well-known 20% solids paste product, this new dispersion has a viscosity comparable to milk and blends into the coating formulation very easily in addition to being considerably easier to handle.

Wax sizes have been used for many years as coating additives for improvement of gloss, smoothness, water resistance, slip, and other sheet properties. Printability of the paper or board has been improved by the use of wax emulsions and dusting at the supercalenders lessened. Recent developments in wax sizes have been in the wax-polyethylene blends. Some of the properties of polyethylene are evident in the final coating when these emulsions are used. Improvement in scuff resistance and ink holdout has been noted in some applications. However, it is still too early to predict the ultimate applications of the wax-polyethylene sizes but we believe these emulsions will definitely find a place in the coating additive field. Surface active chemicals such as sulphonated or sulphated oils, ethylene oxide condensates, and many other types are additives for the coating formulations. A product has recently been developed which combines the action of a defoamer, viscosity controller, and leveler. This material is particularly effective where casein or protein is the adhesive although it has also been used with starch and starch-latex-pigment coatings. Dispersants such as the alkyl aryl sulphonates are sometimes used in coatings to aid pigment dispersion. These materials have recently been improved in several respects. They are supplied as a fine powder of uniform particle size. They dissolve very rapidly to give a clear solution having a pH of 7.5 to 8.0. Because of the chemical composition they are unaffected by most electrolytes.

Other additives to starch coatings which might be mentioned are the viscosity modifiers such as urea-formaldehyde products and dicyanamide and the optical brighteners for improved whiteness.

In conclusion, coating additives have a most important role in the coating field. Without them, your coating would not have that extra plus value so necessary in today's market. However, research and development of coating additives must be intensified. Completely new ideas in this field must be investigated if advancement in coatings is to progress. No doubt, within the next 2 to 5 years, many new products will evolve from the laboratory.

Presented at the meeting of the Gulf Coast Section of TAPPI held in New Orleans, La., May 15, 1959.

Southeastern

The annual meeting of the Southeastern Section was held at Jekyll Island, Ga., on May 8–9, 1959. The host for the meeting was the Brunswick Pulp and Paper Co. R. A. Flick of the Brunswick Co. was chairman of the arrangements committee. Gordon K. Singletary, assistant general manager of the host mill, welcomed the group to Brunswick. In addition to the usual business meeting and technical session, an extensive recreation program was held. A well-planned program was enjoyed by the TAPPI wives with

about 60 taking part. There were over 160 registered at the meeting.

Election of new officers for the coming year was held at the business session. Those elected were as follows:

Chairman: R. R. Chase, Union Bag-Camp, Savannah, Ga.
Vice-chairman: R. A. Flick, Brunswick Pulp & Paper Co., Brunswick, Ga.
Recording secretary: Morton Benjamin, Jacksonville, Fla.
Treasurer: Jimmy Smith, Southern Paperboard Corp., Savannah, Ga.

A tentative schedule of meetings for the 1959–60 year was established as follows: Charleston in September; Rock Hill, S. C., or Charlotte, N. C., in November; Jacksonville in January; Savannah in March; and Jekyll Island again in May. The coming summer meeting of the Executive Committee was scheduled for Savannah for June 12. The treasurer's report showed 333 paid up members in the section and a healthy balance in the treasury. Each of the section officers gave a short review of the work for the year under his jurisdiction.

All three of the papers presented at the technical session were given by Southeastern section members. Two of the papers were entered in the current Section Awards Contest. The first paper entitled "Fiber Fractions and Hand Sheet Properties" was given by Rudolph Schmut of the West Virginia Pulp and Paper Co., Charleston, So. Car. Mr. Schmut's paper dealt with the physical properties of whole pulp, beaten and unbeaten, and the effect of fiber length on these properties. Mr. Schmut's paper is entered in the awards contest. The next paper, also an awards entry, was presented by J. E. Whisenhunt of Sonoco Products Co., Hartsville, S. C. This paper was entitled "Pneumatic Conveying of Purchased Chips." This paper consisted of a brief review of Sonoco's purchased chips program. The final paper of the evening was "Ventilation of Electrical Substations in the Pulp and Paper Industry." This paper will be entered in the awards contest for the next year. It was prepared and presented by R. F. Sorenson of the St. Regis Paper Co., Jacksonville, Fla. Mr. Sorenson discussed the problem of contamination of air on the functions of electrical systems in a paper mill and gave specific methods of minimizing these.

The recreation program was the best yet. On Friday evening the ladies and other guests were taken on a guided tour of the Jekyll Island Museum and then treated to a bingo game with some fine prizes. These activities were timed to entertain the ladies while the men were attending the business and technical sessions. On Saturday morning there were three scheduled events: a deep sea fishing tournament, golf tournament, and a guided tour of the island. The members and guests were about equally divided in attendance at these. The scheduled events concluded with a buffet luncheon at noon. Many of the group spent the rest of the weekend on the island enjoying the beach which is one of the country's best.

Pneumatic Conveying of Purchased Chips

J. E. Whisenhunt

Sonoco's interest in purchasing chips made from sawmill and veneer mill scrap dates back about six years when we found that they were an excellent supplement to our round wood supply. We also noted that their use was in line with our policies on conservation, since they were available without added drain on the forests, and that their cost was no greater than our own cost of chips from round wood.

However, it was not until about 3 years ago that the cost of debarking and chipping equipment was brought within the reach of the small mills which would produce the majority of our chips. It was at this time that we began a survey to determine just how many chips would be available to us as well as the best method to handle them and to evaluate the costs.

J. E. Whisenhunt, Sonoco Products Co., Hartsville, S. C.

We began our studies by writing to several mills in the south that were buying chips to find out how they were handling them, what they thought of their methods, and the cost of their installations. Since our woodyard is located in lowland, below the dam of our 250-acre lake, the shallow water table precluded an economical subgrade installation, and we were looking for one that could be adapted to this location.

Not only was the use of purchased chips new to us, the use of pneumatic equipment to handle them was apparently new to most of the industry in the south. Among the mills that we contacted, none was using pneumatic equipment and most of them said that their present method was an expedient until something better could be developed. The cost of their installations ranged from $30,000 to $100,000.

It was through the trade magazines that we learned that the pulp mills on the West Coast were using compressed air to convey wood chips. They were using it to unload trucks and barges and to distribute the chips to various terminal points. In fact, one sawmill was blowing their chips nearly 1 mile directly into the storage bins at the pulp plant, and from all reports they were doing this at a saving over the cost of trucking. These facts together with our discussions with equipment manufacturers from the west coast, convinced us that the pneumatic conveyor was the best solution to our conveying problem.

Today, we are receiving about 20% of our chips by trucks from sawmills and veneer mills within a 60-mile radius of our plant. Our present plans are to increase this to 30% of our needs by installing facilities for unloading rail cars into our receiving pit. We have set this arbitrary figure at 30% because sawmill production is sometimes limited by bad weather and we feel that to contract for more than this would put our round wood supply in jeopardy. However, we see no problems in getting contracts for this volume because this chip program has been very profitable to the sawmills and veneer mills. In general, the scrap from a thousand board feet of lumber will produce a ton and a half of chips and at the present rate of $7.00 per ton, an average mill producing 20 Mbf. per day could expect a gross income of $50,000 per year from the chips.

When the chips arrive in our yard, they are weighed on our yard truck scales and then directed to the unloading platform. After disconnecting the tractor and anchoring the trailer, the load is tilted to an angle of 50° in about 45 sec. This is a 30-ft. hydraulic platform made by the Air-O-Flex Co. and was installed at grade level for about $9000, including all labor, materials, and equipment.

After the chips are dumped into the receiving pit, a drag conveyor pulls them from the bottom of the pit and meters them into the pneumatic conveying system. Since more horsepower is required to convey the wet chips, the volume of chips being fed into the system must be metered according to their moisture content. This is done with a variable speed drive on the drag conveyor.

The pneumatic conveyor is very simple and flexible. It is driven by a Sutorbelt, positive displacement, high pressure blower and requires 100 hr. to move 22 cords per hr. through an 8-in. pipe. Since this is a positive displacement blower the suction side must be protected with a filter. On the discharge side, we have installed a check valve to prevent the reverse flow of chips which sometimes occurs when there is a plug in the 8-in. pipe line. When this plug occurs, the pressure builds up, relays the fan motor, then the residual pressure in the line is partially relieved back through the fan. This could carry chips back into the fan and cause serious damage to the impellers.

The fan blows under a rotary valve feeder which introduces the chips into the pressurized air stream. From there, the chips are blown about 400 ft. to a cyclone separator on top of the pulp plant. The rotary valve, which is actually the heart of the whole system, was built by the Rader Pneumatics Co. of Portland, Ore. It is equipped with chipper-type knives to shear oversize chips and to rake off any excess fed into the feeder. To date, the only maintenance we have had is to change the knives after each 3000 cords of chips. However, a close watch must be kept for slabs and foreign material which get into the pit from time to time.

We think that our pneumatic conveyor has many advantages, and to us probably the greatest is the savings in first cost. For example, the installed cost of our conveyor was about $18,000, whereas a belt conveyor to do the same job would have cost over $50,000. This equals the total installed cost of all our purchased chips handling equipment. This, however, would not be true on short run installations which are convenient for belt conveyors. Generally, the pneumatic conveyor has its application in long run installations and in short runs where obstructions make mechanical conveyors expensive.

Another advantage is that it is flexible enough to allow us to convey our chips to various parts of our plant, such as the storage silos, pulp plant or to outside storage areas. We can convey them underground, around obstructions, through buildings, or overhead. Most any terminal point is accessible simply by adding the necessary diverting valves and 8-in. pipe to the system.

Although we do not now use outside storage, we hope that with the addition of the necessary facilities and the planned increase in purchased chips, along with a small increase in our own chip production, that we may soon be able to eliminate one shift from our woodyard. If so, we could realize a savings of up to $100,000 per year. Certainly we know of no other type conveyor that lends itself so nicely to such a program at such small cost.

Presented at the meeting of the Southesatern Section of TAPPI held at Jekyll Island, Ga., May 8–9, 1959.

Ventilation of Electrical Substations in the Pulp and Paper Industry

R. F. Sorenson

The reason we install electrical equipment in an enclosed substation in a pulp and paper mill is simply to protect it from the chemicals, fumes, and water that go with the pulp and papermaking process. In short, it is to obtain greater reliability of the electrical apparatus. We defeat our purpose of obtaining greater reliability, however, if we allow the ambient temperature to go too high, or if we allow contaminants to enter the enclosure in our effort to control temperature. The purpose of this paper is to outline the need for controlled temperatures and air treatment as it relates to the substation ventilation and to discuss two approaches that are gaining more acceptance in this field.

RELIABILITY VERSUS AMBIENT TEMPERATURE

Electrical apparatus has heat losses and, if this heat cannot be transferred through the substation walls at a rate sufficient to keep the substation ambient temperature within limits, we must then make provisions to remove this excess heat.

The electrical equipment we use is designed around a 40°C. (or 104°F.) ambient. This does not mean that if we exceed this temperature we will have an immediate failure of the electrical components, but rather it means that the expected or designed life of the equipment will be decreased.

The maximum total temperature that is allowed in a given

R. F. Sorenson, Electrical Engineer, Operations and Maintenance, Pulp and Paper Div., St. Regis Paper Co., Jacksonville, Fla.

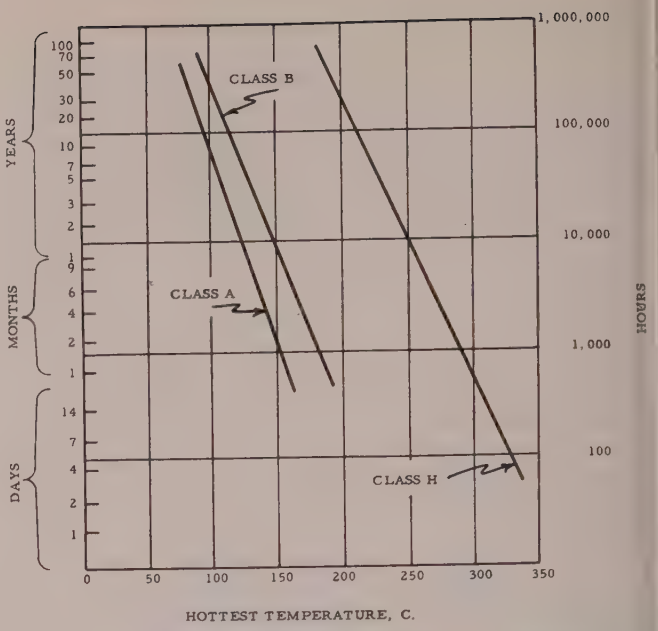


Fig. 1. Insulation life relates directly to working temperature. As a rule, the life expectancy is halved for each 10°C. rise

piece of equipment is usually an arbitrary figure that will allow economical use of materials (lower initial cost) and still give a reasonable life expectancy. These values are standardized by NEMA (National Electrical Manufacturers Association) and are applied to insulations, electrical contacts, bus work, and other components.

Figure 1 illustrates how the thermal life of motor insulation is affected by temperature. It is seen that the minimum life of class A insulation is 10 years at 100°C. If we increase the temperature to 110°C., the life expectancy drops to approximately 5 years, and, if we decrease the temperature to 90°C., we increase the life expectancy to over 20 years. It is generally accepted that each 8 to 10°C. increase in temperature will decrease insulation life to one half of its former value.

Figure 1 points out how insulation life is affected by high ambient temperatures; however, the life and correct functioning of all electrical apparatus we use is always adversely affected by extreme ambient temperatures.

RELIABILITY VERSUS CONTAMINATION OF AIR

When we attempt to control temperature in a substation by external ventilation, we are faced with using air that is contaminated in varying degrees with suspended chemicals, other foreign matter, and corrosive gases. The concentration of contaminants appears to have increased during the past several years. In many cases, we have increased production greatly without increasing appreciably the size of the plant. This often results in higher concentrations of contaminants of the air.

The need for clean air is also greater. Air that was once acceptable is now no longer usable in many cases. This, I believe, is due to several things. The paper industry is using more and more automatic and more precise control systems. This means more complicated and delicate electrical equipment that must be housed in substations. There is also another factor that sometimes goes unnoticed. We have had many advances in the properties of insulating materials. This means that we do not need the same clearances between electrical conductors. Equipment can be built smaller and more economically. Less over-all floor space is required for installation in the mill. This can be a mixed blessing in that we must supply cleaner air when we ventilate so we will not deposit foreign materials that can create conductive leakage paths that lead to electrical failures. Gases and other

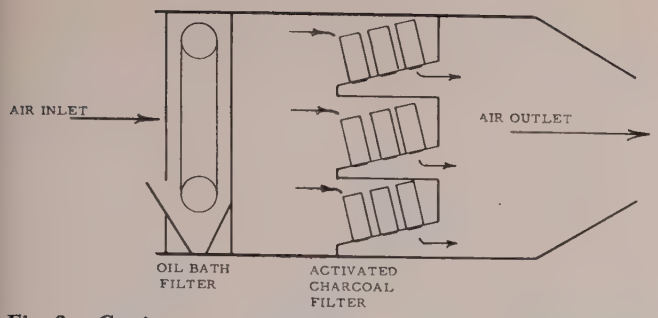


Fig. 2. Canister-type activated charcoal filter installation

contaminants cause rapid brush and commutator wear that affects not only the commutator reliability but can lead to failure of the armature windings due to deposits of carbon and copper in the windings.

CONTROL OF TEMPERATURE AND CONTAMINANTS

We are all familiar with the ordinary mechanical filters, such as the self-cleaning dry filtration and the self-cleaning viscous impingement types of filters. These filters do a good job if applied in a correctly designed system and are properly maintained, but they do have limitations. These filters do not remove gases and extremely small particles from the air. With these mechanical filters, we can expect very fine deposits of lime or salt cake on the equipment in the substation. The search for better methods has focused attention on activated charcoal filters and recirculating closed systems using mechanical cooling (refrigeration) or heat exchangers. These systems attempt to control not only temperature but gases and solids.

The North Western Pulp and Power Co. at Hinton, Alberta, has two of the largest installations of activated carbon filters in the paper industry. This system was put in to remove chlorine dixoide, sulphur dioxide, and hydrogen sulphide from the ventilating air that is supplied to the pulp machine d.c. drive motors and the substation containing the motor generator sets for the sectional pulp machine drive. The substation has a volume of approximately 9000 cu. ft. and contains two 400-hp. motor generator sets; 12,500 c.f.m. is delivered at 1.9 in. of water. The filter system consists of a self-cleaning oil bath (viscous impingement) filter, followed by an activated charcoal filter. The activated charcoal filter uses 650 canister type units, each rated 25 c.f.m. The canisters are fitted into manifold plates as shown in Fig. 2.

The cost of the activated carbon filter was approximately $6900 ($10.80 per cell). This does not include installation cost. The cost of the oil bath filter preceding the carbon unit was approximately $2100. The activated carbon filter was put in operation on Jan. 22, 1958. After 9 months of operation, tests were run on the activated carbon showing an absorption of amines, chlorides, sulphates, sulphides, and sulphur dioxide. Based on the 9-month service, it was predicted that the activated carbon would be effective for approximately $2^1/_2$ years. The canisters are reactivated at a cost of approximately $2.00 each. The average yearly reactivating cost on the above equipment, based on the figures shown, is $520.00 per year. Heating coils are incorporated in this unit to heat the substation during the winter months. A similar installation was made for the d.c. drive motor cooling air on the pulp machine. The installation of these filters has resulted in improved commutation on the d.c. machines, and has eliminated corrosion of electrical apparatus in these areas.

Another approach to the problem of corrosion due to chemicals and gases was taken at the Brunswick Pulp and Paper Co. in Brunswick, Ga. The electrical equipment in a major substation in the bleach plant area was being severely corroded. When the substation was enlarged in their recent expansion, it was decided to recirculate ventilating air in a closed system and remove the heat from the air by mechanical refrigeration.

The system is designed for a maximum substation temperature of 90°F. when the outside temperature is 102°F. The substation has a volume of approximately 4500 cu. ft. and recirculates approximately 17,000 c.f.m. of air. The substation contains two 1000-kva. unit substations and associated motor control centers, four washer drive MG sets and four static rectifier power supplies for washer drives. The ventilating air is circulated through a "throw away" fiberglass filter and then through the colling coils of a "built-up" 40-ton (480,000 B.t.u. per hr.) refrigeration unit. The substation is pressurized to about 1 in. static pressure with 1600 c.f.m. of air brought from outside through a "throw away" fiberglass filter backed up by two panel-type activated carbon filters. The activated carbon filters are rated 800 c.f.m. each. The general layout is shown in Fig. 3. One hundred gallons per minute of water (80°F. maximum) is required for the 40-ton refrigeration unit.

The total cost of this installation was $27,500. The substation is exceptionally clean and shows no evidence of corrosion.

The idea of a heat exchanger was used by the Tacoma, Wash., mill of St. Regis Paper Co. The ground water in that location is such that a water to air heat exchanger is practical. The substation cooling is designed for two 1500 kva. plus four 2000 kva. transformers, associated switchgear and motor control centers, and twelve 20-hp. washer drive MG sets; 36,000 c.f.m. of air is recirculated from the substation through the water to air heat exchanger and back to the substation. When approximately 310 g.p.m. of water at 55°F. is supplied to the unit, they will cool the air from 85°F. to 68°F. The substation room temperature is regulated by controlling the cooling water flow. The system is designed to remove 650,000 B.t.u. per hr. The cooling water is readmitted to the pulp mill fresh water system. The total installed cost of this system was approximately $10,835. The general layout is the same as shown in Fig. 3, except a water to air heat exchanger replaces the mechanical refrigeration equipment.

A third approach, and one that is being used more and more frequently, is the use of packaged air conditioning units, single or in multiple, to remove heat. This approach was used by the East Texas Pulp and Paper Co. at Evadale, Tex., in their original mill. These units have been in operation for about 5 years. The units have water cooled condensers and range in size up to 15-ton units. They report a successful operation of these units. A distinct advantage in packaged units is their ease of installation. When more than one unit is used, a failure of one unit does not represent a complete shutdown of the cooling system.

The extent to which air in substations should be purified, or the extent the temperatures should be controlled, is dictated primarily by cost. This cost must be weighed against the probability of failure of the equipment in the substation

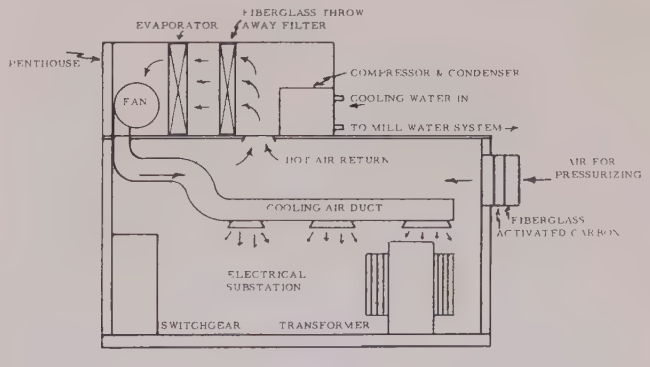


Fig. 3. Recirculating system with mechanical refrigeration

and the cost of the failure, if it occurs, in terms of direct damage and loss of production. The following graphs (Figs. 4 and 5) show the equipment cost for removing heat or ventilating different sized substations under a given set of conditions. The substations considered contained unit substations with associated switchgear and motor control centers sized from 1000 through 5000 kva. Heat losses were assumed to be 60,000 B.t.u. per hr. for each 1000 kva. installed in the substation.

The curve for mechanical refrigeration assumes an outdoor ambient temperature of 90°F. and 60% R.H. Condenser cooling water was assumed to be 85°F. A maximum temperature of 90°F. is to be held in the substation. This design is for removing the heat losses only, and not to cool below outside ambient temperature. The curves for outside air and outside air through charcoal both include an automatic self-cleaning fiberglass filter. The costs shown in Fig. 4 are the equipment costs only and do not include installation costs. You will note that the equipment cost for removing heat by means of mechanical refrigeration in a closed recirculating system becomes comparable with conventional ventilation at the 1000 kva. substation capacity level, or a heat loss of approximately 60,000 B.t.u. per hr. The horsepower requirements, as shown in Fig. 5, indicates that the mechanical cooling will require about ten times the horsepower to power it. Figures 4 and 5 indicate mechanical cooling can be justified on an initial cost basis alone in substations with heat losses up to 60,000 B.t.u. per hr. As the size of the substation and heat loss increases, the premium cost over conventional cooling must be justified by increased reliability of the electrical equipment in the substation. The use of activated charcoal falls in the mid-range and should be considered when the removal of corrosive gases is a problem.

We should minimize our problem by placing the substation in a favorable location in relation to prevailing winds and sources of contamination. We should locate transformers and other noncritical equipment outside of the substation, or in separate substations to reduce quantity of air needed for ventilation, and hence bring such methods of air purification as activated charcoal and mechanical refrigeration into an economical range.

SUMMARY

The reliability of electrical apparatus relates directly to ambient temperature and contamination of ventilating air. It is the mill's responsibility to control both consistent with all economic factors, including lost production, direct damage to electrical equipment and maintenance costs. It is the original equipment manufacturer's responsibility to give the paper industry equipment designed to be compatible with the requirements of our industry. To design electrical equipment entirely around filters would be folly as it is recognized that there will not always be a filtered air supply.

There are many methods of providing clean air; only two are discussed in the paper. The correct one for your mill should be determined by studying carefully what equipment is available and the conditions that exist in your particular case. We must recognize also that equipment must be properly applied and maintained, or it will not do the job.

I feel sure we will all be hearing more about air treatment in our mills. Our management should be made aware of the problems and the work that has been done so they can evaluate the benefits that can be gained.

Presented at the meeting of the Southeastern Section of TAPPI held at Jekyll Island, Ga., May 8–9, 1959.

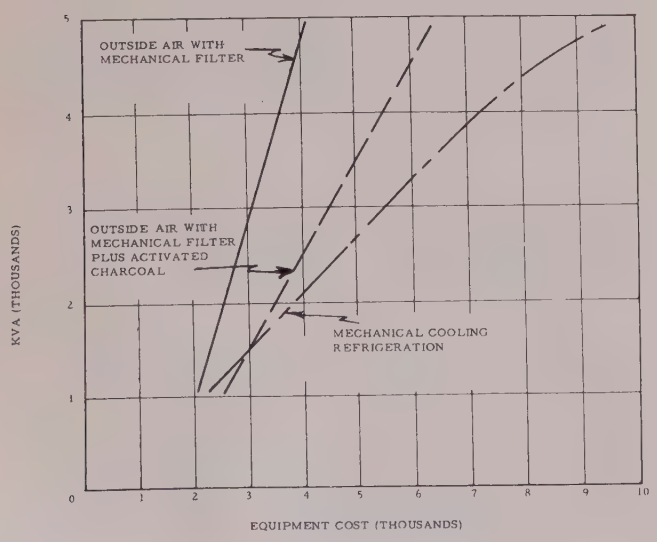


Fig. 4

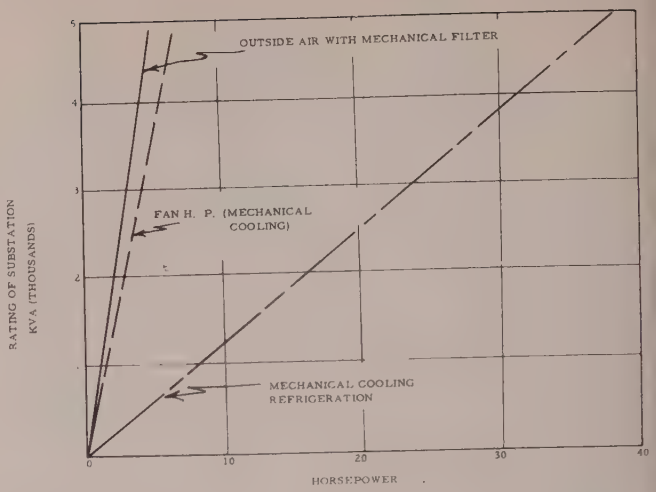


Fig. 5. Horsepower requirements for removing substation heat

Indiana District

The May meeting of the Indiana District was held in the McMillen Room of the Marott Hotel, Indianapolis, Ind., May 19, 1959. A social hour preceded the dinner.

The guest speaker, James Gisondi of the Container Corp. of America's Chicago Design Laboratory, spoke on the subject "Color and Its Effects on Packaging." This discussion was well illustrated with color slides and with samples of cartons. Mr. Gisondi was art director for the General Electric Co. for 14 years prior to joining CCA in 1949.

W. B. Lincoln, Jr., of Inland Container Corp. presented a plaque on behalf of the members to Harris O. Ware for his outstanding work as the Indiana District's first chairman.

The nominating committee composed of S. R. Sutphin, W. B. Lincoln, Jr., M. L. Johnson, and Harris O. Ware presented the following slate of candidates for offices for the 1959–60 period:

Chas. E. Eberly, *chairman*
W. D. Boggess, *vice-chairman* (programming)
John S. Sullivan, *vice-chairman* (membership)
Cushing Roth, *secretary*
Thomas F. Sullivan, *treasurer*

Nominations in addition were requested from the floor and there were none. The election was held and the Secretary was instructed by all members present to record a unanimous vote for the above candidates.

JOHN S. SULLIVAN, *Secretary*

Pacific

The annual joint meeting of TAPPI and the APPMSA was held at Gearhart, Ore., May 21, 22, and 23. The theme

of TAPPI's part of the program was "Multiple Water Use." The session beginning at 8:30 a.m. and chairmanned by Norval Magnusson was very well attended and testified to the interest and timeliness of the subject matter. The papers given and their authors are as follows:

1. "Water Quality Criteria as They Relate to Beneficial Water Uses," by Vinton M. Bacon, executive secretary, Northwest Pulp and Paper Association, Tacoma, Wash.
2. "Legal Aspects of Multiple Water Use," by Daniel C. Smith, Weyerhaeuser Timber Co., Tacoma, Wash.
3. "The Conservationist's Viewpoint," by L. C. Binford, vice-chairman, National Executive Board, Izaak Walton League of America, Portland, Ore.
4. Industries' Use of Bio-assay Methods—"Bio-assay Techniques Used in Tests at Simpson Paper Co.," by Dwayne J. Clark, Simpson Paper Co., Everett, Wash.; "Bio-assays—A Tool of Industry," by Norman S. Lea, Scott Paper Co., Everett, Wash.; West Tacoma Newsprint paper by James Cooper.
5. "Relationship of Fisheries Statistics to Industrial Wastes," by Roger Tollefson, Consulting Biologist. Paper read by Dick Petersen, Rayonier Marine Laboratory.
6. "Accomplishments of Mills on Water Quality Control," by S. K. Pratt, St. Regis Paper Co., Tacoma, Wash.

Following the morning program, the annual business meeting was held and the election of officers for the 1959–60 year was accomplished. Elected chairman was Norval Magnusson; vice-chairman, P. T. Dickie; secretary-treasurer, Robert H. Smythe.

A round-table session in the afternoon, moderated by Donald G. Felhous, discussed "Technical Sales and Industry's Needs." The various departments represented were:

Purchasing—J. D. Morgan, Oregon Pulp & Paper Co., Portland, Ore.
Engineering—C. L. Carns, Weyerhaeuser Timber Co., Longview, Wash.
Technical Sales—Meder Johnson, James Brinkley Co., Seattle, Wash.
Technical Department—John Dunkak, Puget Sound Pulp & Timber Co., Bellingham, Wash.
Operating Superintendents—James Palmer, Crown Zellerbach Corp., Camas, Wash.

The International Brotherhood of Migratory Peddlers, Waiting Room no. 1, put on the "Wake-em-up" breakfast show Saturday morning to a full house. Initiated as an honorary member in the Brotherhood was Harold W. Bialkowsky, director of research, Weyerhaeuser Timber Co. Mr. Bialkowsky is also a past chairman of Pacific Section-TAPPI and one of the first to receive a Doctor's degree from the Institute.

Saturday evening was highlighted by a social hour around the swimming pool and a gala banquet at which Chairman Paul Billington reigned as toastmaster. Dancing and a splash party were the final events on a very successful convention.

A total registration of 535 persons made this one of the largest of the Gearhart meetings. Women made up a good part of the attendance (207) and contributed to the spirit of the meeting. An interesting feature was the fact that in excess of 90% of those attending were preregistered.

ROBERT SMYTHE, *Secretary-Treasurer*

Water Quality Criteria as They Relate to Beneficial Water Uses

Vinton W. Bacon

IT IS my understanding that this is the first time that the subject of multiple water use has been included as an entire session at one of your principal annual meetings. I know that I speak for each of the eight participants in saying that we are honored to speak to you on this occasion on some of the aspects of water resource uses and management.

Although this was billed as a paper on beneficial water uses, it might be more accurately titled "Water Quality Criteria As They Relate to Beneficial Water Uses." As this is one of the first meetings on the subject of water, these may be strange terms to some of you. Because they are broad terms, I will not attempt to define them in the classical manner, but rather to develop their meaning and significance by discussion and illustration. Please bear in mind, though, that I am speaking of water outside of, not within, cities and plants.

VINTON W. BACON, Executive Secretary, Northwest Pulp and Paper Association, Tacoma, Wash.

STEPS IN WATER QUALITY MANAGEMENT

Although greatly simplified, rational management of quality in either fresh or salt water may be accomplished through three logical steps:

1. By geographical areas, determine and enunciate present and future beneficial water uses which are to be preserved and protected.

2. Determine and adopt water quality criteria to protect these uses, but which at the same time allow maximum use and re-use of the waters.

3. Establish monitoring to measure maintenance or meeting of the water quality criteria.

There will be no discussion of the third step, monitoring, as this should be a matter of mere mechanics.

BENEFICIAL WATER USES

A listing of major beneficial uses of water is given below. It should be recognized that these water uses are not listed in the order of economic importance, nor in the order of increasing or decreasing water quality requirements. They are:

1. Domestic water supply
2. Industrial water supply (including cooling water)
3. Agricultural water supply (irrigation)
4. Stock and wildlife watering (including refuge for water fowl)
5. Propagation of fish and other aquatic life
6. Shellfish culture
7. Swimming and bathing
8. Boating and esthetic enjoyment
9. Water power and navigation
10. Waste dilution and dispersion

It is not necessary to emphasize to this group that all water uses are not compatible from the quality point of view. Because of bacteria, unregulated discharges of domestic sewage can render a river unsuitable as a source of domestic water supply or for swimming. Discharges of organic waste from communities or industries can so consume dissolved oxygen from the receiving water as to make it untenable for fish. Diversions for irrigation can conflict with fisheries resources due to increased temperatures, mineralization, and turbidity of return drainage waters. Oil discharged from countless outboard motors might not harm lake waters for boating, but it could render them unattractive for swimmers and harmful to fish life. In one area with which I am familiar, the principal controversy was pollution of ranchers' domestic water supply by a state fish hatchery. All waters cannot be maintained in their so-called "natural" state—which really means primeval state—without excluding all other beneficial uses, including fishing itself. I am sure that many other examples of conflict among water users suggest themselves to you.

Although listed last, waste dilution and dispersion is an extremely important beneficial use. In fact, I know of no laws that by expression, implication, or regulatory procedures do not make such provision.

In both kind and number the beneficial uses of water vary from area to area. Further, within areas uses change from time to time. In most cases these changes are very gradual, and social and technological developments can keep pace. But the change can be abrupt as in the case of Grand Coulee and Shasta Dams where the uses of water upstream changed

rapidly from anadromous fisheries to irrigation and power developments.

Someone—or some authority—finally has to make the decision as to the relative importance of the various water uses being, or to be, protected. In the past, because municipal and industrial wastes were regarded as the principal variable in the water quality control picture, this decision has been made by pollution control authorities, who have exercised, either consciously or unconsciously, this grave responsibility. In realization that impoundments, irrigation, land use, minimum flows, and other factors can seriously affect water quality, the trend recently is to delegate the over-all state planning and coordination of water resources development to an over-all state water department, leaving the responsibility for controlling waste discharges to the pollution control authority. In either event, public hearings are used extensively, where it is up to the individual water user to make the best case he can. The hearing board asks for the best analyses various water users can present, so that their decisions will be equitable. It should be remembered that such hearings are not—or most certainly should not be—sounding boards. To function equitably, such hearings should be competent, fact-finding sessions.

NORTH PUGET SOUND WATER-USE HEARING

A good example of the hearing process to determine water uses of a marine area occurred in Bellingham, Wash., on March 12 of this year. In announcing the hearing, the Washington Pollution Control Commission stated it recognizes that the salt waters of Northern Puget Sound are utilized for a number of useful purposes and that there is a need to establish receiving water quality criteria which are based on the uses and the resource values of these waters. It further

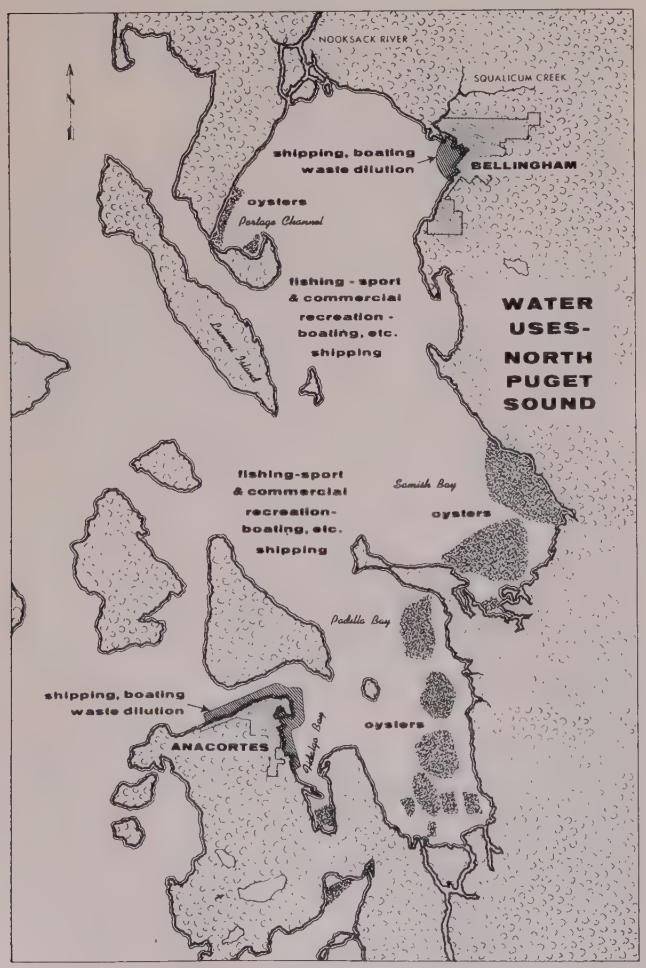


Fig. 1

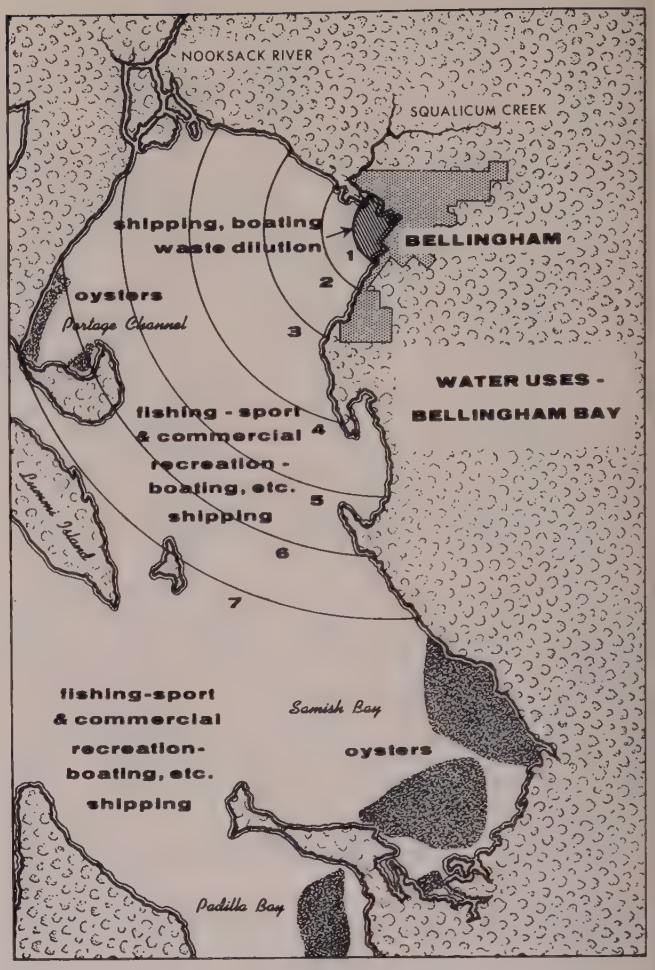


Fig. 2

stated that information is needed on the extent, seasons, and values of the various uses of the areas to enable the Commission to initiate a program to maintain suitable conditions of water quality so that maximum and sustained resource benefits may be derived. Thus the Commission recognized the concepts of multiple and maximum water use and asked for expressions thereon. By using the material submitted by Ericsson (*1*) of the pulp and paper industry of the area, I am going to show, in part, what they thought were reasonable uses of the marine waters.

On the map of the area (Fig. 1), we show what appear to be reasonable uses of water resources in this area. In a strip around Anacortes, and within a distance of approximately 3600 ft. of the Bellingham waterway (as shown by the cross-hatched areas), we believe the maximum and sustained resource benefits will be derived when the principal uses are marine transporatation, boating, and waste dilution and dispersion. At the periphery of these areas, migration of salmon and steelhead should be a principal use. Beyond the periphery, the major water resource benefits are: fish migration, propagation, and grazing; sport fishing; commercial fishing; recreation (boating, swimming, skiing, etc.); shipping and marine transportation.

At distant points, such as Portage Channel, and Samish, Padilla, and Fidalgo Bays, oyster culture should be added to the list of uses. Insofar as the information was available, the locations of the oyster lands in these areas are shown.

On this map, we have indicated what we think are legitimate and necessary waste dilution and dispersion areas for the communities of Anacortes and Bellingham and their industries. The reasonableness of the size of these areas is apparent when compared to the total water area and volume of North Puget Sound as is developed on the next two figures.

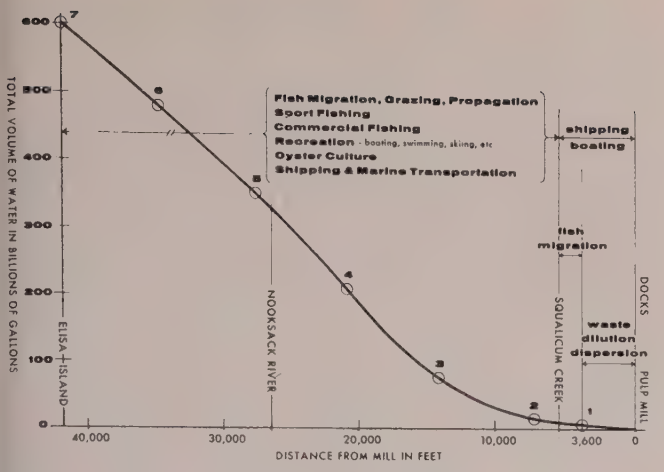


Fig. 3. Volume of water for various uses in Bellingham Bay

Figure 2 is a chart of Bellingham Bay which was used to compute the volume of water resources suggested for the various uses. Using Puget Sound Pulp and Timber Co. docks as a center, arcs were struck as shown. The first arc (no. 1) of about 3600-ft. radius defines the area of dilution and dispersion as suggested in the previous figure. The last arc (no. 7) touches Lummi Island, crosses Eliza Island, and includes the most northerly part of Samish Bay. By means of areas and depths, the volume of water at low tide within each ring was computed. Please note the arc numbers.

Figure 3 shows a plot of the total volume of water within the arcs and for the various water uses. To orient it with the last figure, the curve reads from right to left. The various arcs are numbered along the curve. On the extreme right is the location of the pulp mill. Within the first arc, which defines the area within which waste dilution and dispersion are recognized uses, the volume of bay water is $2^1/_2$ billion gal. This very large quantity of water affords an initial dilution of 75 to 1 for stabilizing the mill effluent and other community and industrial wastes. But this $2^1/_2$ billion gal. is minute when compared to the nearly 600 billion gal. of bay water between arcs 1 and 7 which are being protected for other uses, including fish and oyster culture. The curve vividly depicts this relationship in quantities in that point 1 is hardly off the graph base line.

The argument will be advanced that wastes diluted and dispersed in the waterway will move beyond arc 1. Of course, this is absolutely true! But by the time it reaches this arc the material has become so diluted and stabilized that other water uses are not unreasonably and adversely affected.

In summary, the industry felt that its recommendations for areas and water uses were sound, equitable, and safe because:

1. The waterway adjacent to the city of Bellingham and the mill is zoned industrially and is not a fishing area of economic consequence because of upstream uses and development, and low summer flows.

2. Waste dilution and dispersion is recommended as one of the principal uses for only a small fraction of the bay. This seemed more than equitable when the value of the industrial products and the needs for economic waste disposal for both the city and its industries are taken into consideration. To do otherwise would seem to ignore the present and future economic well being of the entire area.

3. Fisheries interests (salmon, steelhead, bottomfish, and oysters) would not be unreasonably or adversely affected if scientifically supportable water quality criteria are specified.

Although a marine area has been used as an example, the same principles and procedures apply to fresh water areas, such as rivers, lakes, reservoirs, and storage pools.

WHAT IS POLLUTION? WHAT IS MAXIMUM SAFE WATER USE?

The foregoing has been a discussion of what was presented as step 1 in water quality management—determine by areas, then enunciate present and future beneficial water uses which are to be preserved and protected.

Now we are down to step 2—determine and adopt water quality criteria to protect these uses, but which at the same time allow maximum use and reuse of the waters.

In this case, I borrow from material which was presented by Forman (*2*) at the Bellingham hearing.

One of the first points to be clarified in considering this subject is to resolve the question "What is pollution?" This is entirely possible, but it is not easy. Unfortunately, many people who have not studied the subject scientifically have the impression that putting anything in the way of a waste material into public water is pollution. They ignore, or are unaware of, the fact that while some wastes are toxic, many are nontoxic. They ignore, or are unaware of, the very real capacity of water to receive and assimilate wastes and return to its original condition without suffering damage in this very natural process. Lack of understanding on this question can be one of the most serious impediments to development of a sound water quality management program.

What then is pollution? In legal language a court in Colorado has defined it as follows:

> "For the purpose of this case, the word 'pollution' means as impairment, with attendant injury, to the use of water that plaintiffs are entitled to make. Unless the introduction of extraneous matter so unfavorably affects such use, the condition created is short of pollution. In reality, the thing forbidden is injury. The quantity introduced is immaterial."

Other states have recognized waste dilution and dispersion as a legitimate practice insofar as such waste materials do not unreasonably and adversely affect other beneficial uses of water. For instance, one state defines pollution as:

> "Pollution means an impairment of the quantity of the waters of the State by sewage or industrial waste to a degree which does adversely and unreasonably affect such waters for domestic, industrial, agricultural, navigational, recreational or other beneficial use."

Although Washington law does not define pollution as such, the Pollution Control Act does embody the principles of multiple and maximum use of our waters. In part, Section 1 provides that it is the public policy of the State of Washington to maintain the highest possible standards to insure the purity of all waters of the State consistent with public health, public enjoyment, protection of wild life, and the industrial development of the state. Further, Section 13 declares that the state shall determine what qualities and properties of water shall indicate a polluted condition deleterious to the lawful occupation in which the state's waters may be lawfully used.

Thus, it is apparent that the discharge of a waste *per se* is *not* pollution. In both the legal and practical sense, pollution occurs only when the *concentration* of a waste is such as to adversely and unreasonably harm other water uses. Anything below these concentrations *does not* constitute pollution.

Let me illustrate this important relationship between concentration and pollution with a few examples. A few grains of salt in a glass of water does not interfere with your enjoyment of drinking it. You don't know it is there, but if you put a tablespoonful of salt in a glass of water you have surely spoiled it for drinking. So, at what concentration does pollution begin? Now, some people enjoy a cocktail before dinner. Drinking enough straight grain alcohol can kill you, but a little diluted in a cocktail is said to be rather pleasant! So, again, the concentration is important. Our furnaces, our cars, and even our lungs are putting out many tons of carbon dioxide into the air right here in the city

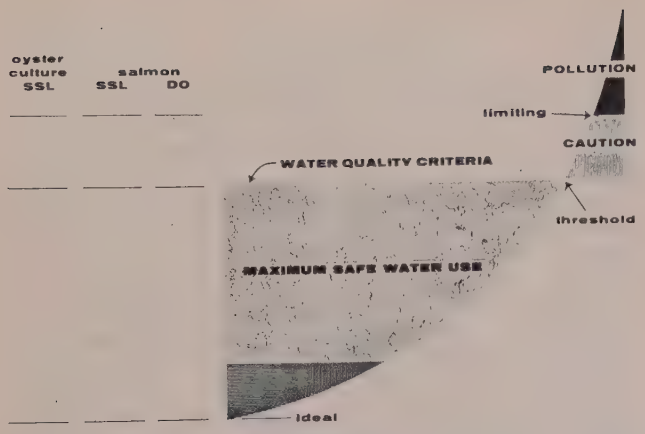


Fig. 4. What are water-quality criteria which allow maximum safe use?

today. We don't suffer from this and we don't usually think of it as pollution. However, if it were concentrated in this room we would die of suffocation. What do we do in our homes, where the furnace gases would be harmful if discharged in the house? We run a chimney out the roof where these gases are *diluted* and *dispersed* and generally do not bother anyone.

Put a substance—any substance—into the waters of a region and you have potential pollution—pollution in the sense that, if sufficiently concentrated, the substance can adversely affect such waters for one or more of its established uses. Yet, any substance, when adequately diluted, can also be rendered harmless to those same uses. A waste discharged, for example, into a tiny stream may result in gross pollution to all established uses for that stream. Identical amounts of that same waste introduced into the Columbia River or Puget Sound may cause no adverse effects at all.

From the above, it is apparent that a rational definition of pollution will depend (1) on the water uses in a particular area and (2) on the concentrations of extraneous material which can exist without adversely affecting the uses. These concentrations are frequently called "water quality criteria."

What are these criteria that allow maximum safe-use of water, that is, pollution-free use? This is illustrated on Fig. 4. The curve as it ascends from left to right represents increasing concentrations of waste material in the water. Now, every water use (such as fishing, oyster culture, etc.) imposes two values on each of its water quality criteria. First, there is a "threshold" value, or concentration below which the water use is not damaged to any significant degree by waste materials. Below this value is the area of maximum safe use of water. Second, there is a "limiting" value, or concentration above which the water use is to all practical purposes inhibited or destroyed. Above this value, pollution occurs. Between the threshold and limiting values is a caution or questionable zone. Concentrations in this zone can be tolerated safely for limited periods of time, and they warn us that we are approaching the upper allowable limit for pollution-free use of the water.

To get a little clearer picture of what we are trying to illustrate on this chart, let's return for a moment to the example of salt in drinking water. Practically no salt (or only a trace) as represented by the lowest point on the curve would be the optimum or ideal level, but anything between this point and the threshold value is still a perfectly satisfactory drinking water. Between the threshold and limiting concentrations is the caution range. And, of course, if the salt concentration gets above the limiting value, the use of the water for drinking would be inhibited or destroyed. That is, it would be polluted as far as this particular use of water is concerned. Sea water, with over 30,000 p.p.m. of salt would be well in the pollution area for purposes such as drinking, but safely in the safe-use area for shipping, boating, and certain fishing activities.

On the chart, I have shown salmon and oyster culture as water uses, with the spaces below to indicate limiting, threshold, and ideal values for water quality criteria. Utilizing scientific and research data and vast practical experience which are available, it should be possible to set water quality criteria to obtain *maximum safe-use*. These values are represented by the horizontal line marked "water quality criteria." It should be emphasized that setting of standards is a highly technical task and certainly not one which can be equitably achieved through bartering and horsetrading. Likewise, it should be emphasized that if not properly done dire consequences can result to a state's over-all economy. As an illustration of this, it certainly is possible to set criteria at the level depicted by the area in the very lower left-hand portion of the chart. Certainly such level would be *safe*. But it would be equivalent to practically eliminating all of the beneficial uses of water other than esthetic. Represented graphically, such recommendations would shrink the maximum safe-use to practically nothing.

Numerical criteria have not been shown on the chart as there are honest differences of opinion as to what should be specified for these uses. In an effort to reconcile them, the State of Washington has retained a board of scientific consultants as these differences must "yield to scientific determination."

Summarizing to this point:

1. Present and potential beneficial uses of water must be determined. They control criteria.
2. Water quality criteria are those limits of concentrations within which water uses are not unreasonably or adversely affected. There is a threshold value and a limiting value.
3. Waste discharges *per se* are not pollution. Pollution occurs only when water quality criteria are exceeded.

FRESH WATER CRITERIA

I'd like to illustrate criteria for fresh water uses.

Assume that the proper authorities have determined that the uses of a river to be protected shall be irrigation, fishing, and bathing. Figure 5 shows water quality criteria for protecting these uses in this hypothetical case.

For irrigation, the dissolved minerals criteria are assumed as 700 p.p.m. threshold and 1500 p.p.m. limiting. As we all know, crops do not do well if the water becomes too "salty."

The corresponding irrigation water criteria for sodium (Na) is 40% threshold and 60% limiting, the percentage referring to the ratio of Na ions to the total of all cations (Na plus Ca plus Mg plus K). Ca and Mg are the constituents of hard water; Na of soft. The different effects of the two on land were described by Scofield (*3*), "Hard water

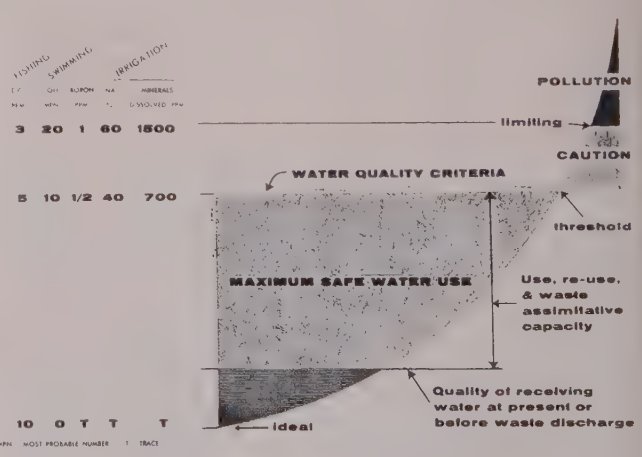


Fig. 5. Demonstration of water quality criteria

makes soft land and soft water makes hard land." High Na concentrations are undesirable.

Corresponding criteria for boron are assumed as 0.5 p.p.m. and 1 p.p.m. Although essential in trace quantities for good growth, boron is usually injurious above 1 p.p.m. to the boron-sensitive plants such as citrus, apples, pears, walnuts, and pecans. Even for tolerant crops (sugar beets, alfalfa, carrots, lettuce, asparagus), the limiting value should not exceed 3 p.p.m. very often or for prolonged periods.

For bathing, the criteria used in the illustration are as most probable number (MPN) of coli bacteria of 10 per ml. (1000 per 100 ml.) threshold and 20 per ml. (2000 per 100 ml.) limiting. Although coli bacteria are not pathogenic (disease formers), they originate in the intestines of warm-blooded animals. Therefore, they are taken as indicative of human contamination. State requirements for bathing waters vary considerably, from a low of 50 per 100 ml. to a high of 3000. The Pacific Northwest Pollution Control Council, an unofficial group, recommends an objective for bathing of 240 per 100 ml.

For migrating and feeding fish the illustrative criteria for dissolved oxygen are given as 5 p.p.m. threshold and 3 p.p.m. limiting. From recent scientific work, M. P. Shepard (*4*) of the Department of Zoology of the University of Toronto concluded that within their first year most young speckled trout can withstand oxygen concentrations down to 1.9 p.p.m. The Fisheries Research Board of Canada (*5*) observed that salmon, in addition to bottomfish, were present on Barkley Sound in dissolved oxygen concentrations of 1 p.p.m. (11% of saturation). But at the other end of the D.O. spectrum, recent work by Alderdice (*6*) on chum salmon indicates that critical levels ranged from about 1 p.p.m. in the early stages of egg development to over 7 p.p.m. shortly before hatching.

It should be emphasized that this hypothetical case has been oversimplified, and the figures are for illustrative purposes only. In actual case, criteria in addition to those shown would very likely be given for each water use. Additional or different water uses would have to be considered. Seasonal variations in water use would dictate seasonal variation in water quality criteria. If during certain seasons the waters are not used for bathing, or if fish can safely tolerate lower dissolved oxygen concentrations, a waste discharger would be allowed to reduce his treatment and disposal costs by making greater use of the waste assimilative capacity of the river. Soil type, crops, drainability, precipitation, cold-water species of fish, warm-water species, extent of bathing use, and many other factors and variables must be considered in arriving at criteria to make maximum use of our receiving waters.

Note the horizontal line in the lower portion of Fig. 5 which is marked "quality of receiving water at present or before waste discharge." According to Sylvester (*7*), the yearly weighted averages for the Columbia at Maryhill for the period 1952–56 were:

Dissolved solids	116 p.p.m.
Na	24%
Boron	(Not reported)

These are well below the criteria used in the illustration indicating in this example much use, reuse, and waste assimilative capacity of the waters at that point.

The same is true for the other two criteria according to the U. S. Public Health Service (*8*). At Bonneville, the average D.O. is in the vicinity of 10 p.p.m. and coliform count of approximately 51 per 100 ml.

Most waters of the Northwest are of similar high quality.

For future reference to this figure, I'd like to ask you to keep in mind the "ideal" point and the small area in the lower left-hand portion of the chart.

You will note that I have not dwelled at length or presented extensive tables on numerical water quality criteria for the various water uses. These are superbly presented in Publication No. 3 of the California Water Pollution Control Board (*9*).

USE CHANGES QUALITY

The question is frequently raised "Why allow any change from our present good quality?" Why not status quo? This ignores the simple fact that practically every water use results in change in quality—and usually in a downward or degradation direction.

In passing through our communities, waters pick up both organic and mineral matter. Although biological treatment of wastes will stabilize the organic matter and chlorination will kill bacteria, the total dissolved solids may be expected to increase by 100 to 300 mg. per l. (*10*), corresponding to 70 lb. per capita per year. Such new compounds as detergents are neither readily stabilized nor removed by the commonly used methods of sewage treatment.

Agricultural use represents a major impact on water quality. Even in the absence of irrigation, the cultivation of land affects the quality of surface and subsurface waters. These effects are intensified when irrigation is practiced.

As many of you know, in irrigation, the soil must be well drained so that the plant roots do not become water sick and so that salts do not accumulate at the soil surface. Excess water must be applied to remove the salts not used by the plants. Via drains, waste-ways, and ground water, much of this excess water is returned to the receiving stream. Of the applied water, 20 to 60% may be returned. It is more highly mineralized, has different physical properties, and has often picked up remains of fertilizers, insecticides, and other agricultural chemicals.

At the present time water diverted from the Colorado River below Hoover Dam for irrigation in Arizona and California has a total dissolved solids content of about 700 p.p.m. Estimates vary considerably as to what changes in quality will result from development of the Upper Colorado River Basin Project for irrigation. Hill (*11*) estimates that the average total dissolved solids in the water available for diversion below Hoover Dam in the future will exceed 1140 p.p.m. as a result of upper basin development.

Hill uses another example to demonstrate that return irrigation waters pick up salts. Between Glenwood Springs and Cisco, the salt content of the Colorado River increases three times (from 0.305 to 0.918 tons per acre ft.) due mostly to irrigation along the Colorado and tributaries entering between the two points.

Sylvester (*7*) records the changes between 1910 and 1954 in quality of the Yakima River at Prosser in September. During this period, 240,000 additional acres of land were placed under irrigation, and industry and communities increased equivalent to a population of 216,000. On a weight basis, this growth resulted in the following percentage increases in constituents:

	Per cent
Alkalinity	610
Hardness	435
Sulphate	100
Dissolved solids	400
Calcium and magnesium	450
Sodium and potassium	390
Silica	775
Chlorides	166
Nitrates	6250

He also noted that the temperature rise in August between Thorp and Richland (160 miles) is 17°F. (54 to 71) due largely to irrigation return flows.

Industries in their use of process and cooling water change the physical, chemical, biological, and even radiological characteristics. Impoundments for power and flood control alter water characteristics. In this case the changes can be toward improved quality through reduction in temperature

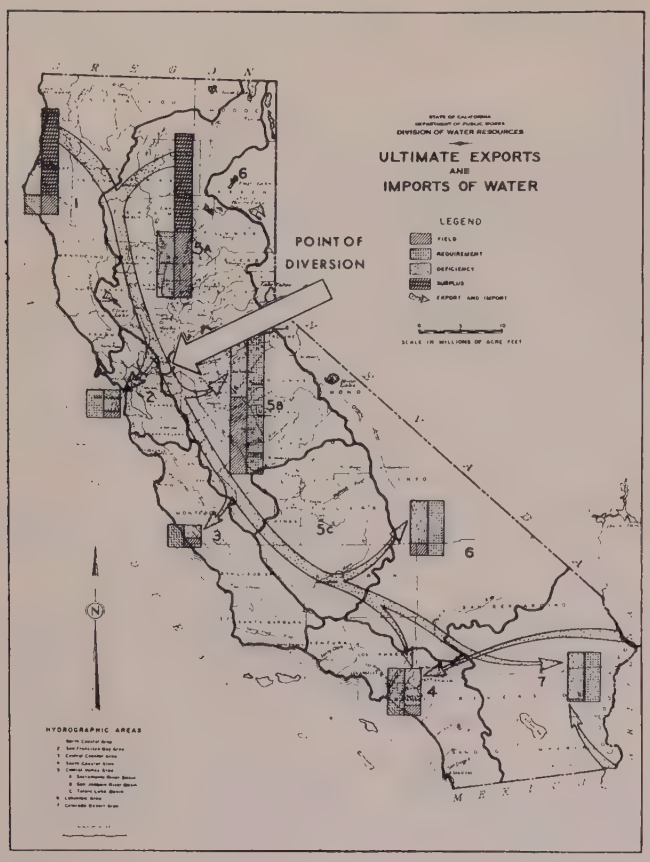


Fig. 6

and turbidity. Recreational use can also degrade water through trash and debris, bodily contact, and oil and gas from outboard motors and spills. This latter use may not appear significant to the sportsman, but it does to the water supply superintendent who is charged with the responsibility of delivering a safe and potable water supply.

The moral in these examples is that water use is accompanied by quality changes. The only way to avoid the change is to prohibit further expansion of industry, communities, irrigation, and other aspects of our economy. The only way to go back "to the good old days," as some unthoughtfully suggest, would be to eliminate our present development. This would be trying to return to the ideal point on the previous chart by eliminating the water use represented by the area in the lower left-hand portion of the chart.

ARE CRITERIA FOR MAXIMUM USE PRACTICAL?

If we conclude we are going forward, not backward in our economy, does the procedure of setting water quality criteria to permit maximum safe use make sense and is it practical? Some areas are already coming to it. California and the Ohio River areas are good examples.

As many of you know, Northern California is blessed with a surplus of undeveloped and unused water. Southern California wants some of it—provided the quality is right. As shown in Fig. 6, the point of diversion would be from the Sacramento River in the vicinity of the Sacramento-San Joaquin delta. The accompanying table shows water quality considerations.

The first column shows the limits for water export as recommended by a board of water quality consultants and as adopted by the Department of Water Resources (*12, 13*). The second column shows present quality of the Sacramento River in terms of median values at Sacramento (river mile 62.5) for the 1951–56 survey period (*14*). Harkening back to Fig. 5, the difference between the two columns is the use capacity being reserved for the northern part of the State.

Water Quality in California Water Plan

Item	*Limits for export*	*Sacramento River at present*
Total dissolved solids, p.p.m.	400	110
Electrical conductance (Ec x 10^6 at 25°C.)	600	176
Hardness as $CaCO_3$, p.p.m.	160	62
Sodium percentage, %	50	28
Sulphate, p.p.m.	100	13
Chloride, p.p.m.	100	8
Fluoride, p.p.m.	1.0	
Boron, p.p.m.	0.5	
pH Value	7.0–8.5	
Color, p.p.m.	10	
Other constituents	USPHS	

The board of water quality consultants (*13*) concluded that "the limits set forth will permit full agricultural development in northern California, provide for greatly increased population in that area, and allow the establishment of all industries required for the support of that population."

A method of apportioning the capacity of a stream to assimilate chloride—also applicable to many other pollutants—was suggested in March, 1957, by the Ohio River Valley Water Sanitation Commission (*15*). First step recommendation would be establishment of a standard that the chloride concentration at any point on the main stem never exceed at a monthly average of 125 p.p.m. at the most probable minimum monthly average 10-yr. flow, and that the maximum concentration never exceed 250 p.p.m. Figure 7 shows that the existing chloride load at the most critical point of use is just about in balance with the "average" standard recommended. Should new chloride loads necessitate cutbacks, they would be imposed only on industry on one of two proportionate discharge systems. Although numerical standards were not adopted, the Commission implemented a chloride-control program on Sept. 12, 1958, based on safeguarding the quality of the waters of the Ohio River and tributaries and of securing maximum utilization of

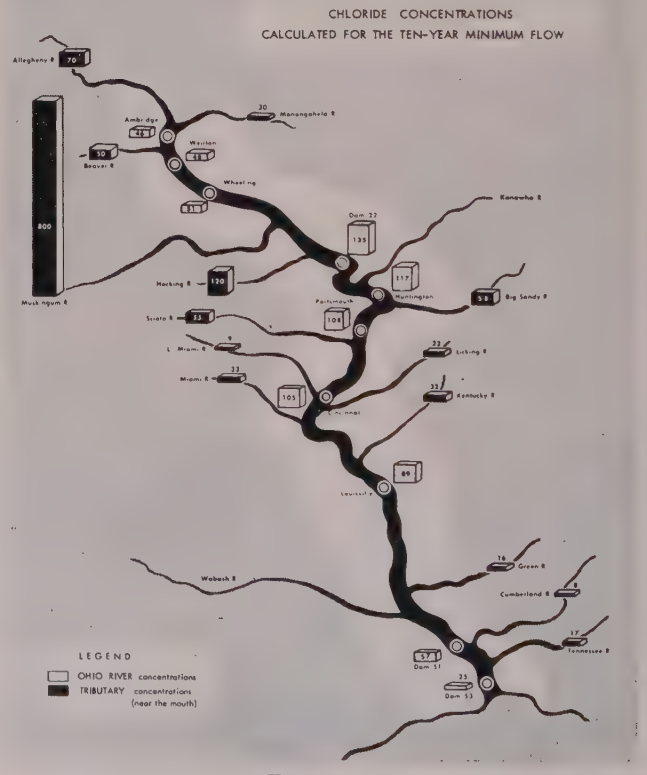


Fig. 7

the capacity of those waters for assimilating chloride waste discharges (*16, 17*).

It seems to me that these two cases involving extremely complex water management problems demonstrate the application of water quality criteria to obtain maximum safe use of waters. Returning to Fig. 5, what is the relationship in time of these two areas to the Northwest. The Ohio area, and much of the East, is in the "pollution" area. They have to come back down the curve, using not only planning but correction in the process. And they are doing so! In the Southwest and California many of the waters, particularly those in areas of heavy irrigation, are practically spent—that is, quality-wise. They are at the water quality criteria line. In the Northwest we are far down in the maximum safe-use area, in most areas. What a heritage of potential safe use we have!

Maximum comprehensive development of our great river basins and waterways for all beneficial uses has long been recognized as a guiding principle in the water policies of the United States. It would be difficult to find a more precise, clear-cut affirmation of this principle than in the words of President Theodore Roosevelt in 1908:

> "Every waterway should be made to serve the people largely and in as many different ways as possible.....Every stream should be used to its utmost....Each river system, from its head waters in the forest to its mouth on the coast, is a single unit and should be treated as such" (Senate Document No. 324, 60th Congress, 1st Session).

The same principle applies to marine waters.

To get maximum basin and water development, Marshall Dana (*18*), keynoter at the Regional Water Resources Conference at the State College of Washington in November, 1958, admonished all to deemphasize their differences and controversies. He suggested cooperation, coordination, self-examination, and a fierce determination to do the job ourselves, that is, without outside interference. As far as water quality is concerned, we in the Northwest are far ahead of the game. Through logical planning and research kept in balance with our needs, we can proceed to make maximum safe use of our waters.

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Multiple Water Use—the Conservationist's Viewpoint

L. C. Binford

THERE is no phenomenon of American society so astounding as that ability of a convention committee to reduce a freedom-loving people to an enslaved forum such as we have here this morning.

You, the captive audience, under the device of an expense account, are here to be exposed to the brainwashing of a few speakers who perform for your benefit for no other reason or consideration than that your convention committee, by slightly fanning our ego, inflamed our crusading, missionary zeal.

Now for some of you who have not had experience in the technique of securing convention speakers, let me quote from the letter that was used to entrap me.

But first I must explain that the letter was signed by an old friend—very chummy, "Sincerely, Bob." At first I thought it was from Bob Holmes but then I remembered we had not been writing since last September.

Then in very conservative type I noted it was on the letterhead of Western Kraft Corp. Now listen to Bob's line: "Dear Jack: An unusual opportunity to present the conservation viewpoint on multiple water use to an important group of industrial water users presents itself in the attached tentative program for the Pacific Coast section of the Technical Association of the Pulp and Paper Industry. From 200 to 300 or more pulp mill superintendents, technical directors, technicians and assistants, chemical engineers, etc. attend these meetings.

"In the last analysis these are the men on the production 'firing line' in the pulp industry who, in day to day operation of a mill, can do more than any other group to minimize pollutional effects on a given mill's effluent on receiving waters. The general session on multiple water use has been designed to broaden their viewpoint on this problem.

"Would you consider taking on the assignment? Your thorough knowledge of the subject, your identification with the League and with the water resources board, and your reputation with industry generally as a man who presents facts reasonably, clearly, and effectively makes you the ideal person for this job."

Now that last paragraph got me. For three years I had been going around the state talking about water resources and I thought I had the support of the people of Oregon. Mark Hatfield thought so and put me back on the Oregon Water Resources Board. Within a few days my

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ego was deflated by having all of the Democrats in the Oregon Senate except two, vote against my confirmation.

Naturally I was pretty low, and that line, "your reputation with industry generally as a man who presents facts . . ." really sold me. I accepted, in the words of Bob Beatty, "the unusual opportunity."

Oh, yes, Bob said to me, "TAPPI as a group is strictly professional and nonpolitical. This does not mean, however, that you should avoid discussing political aspects of multiple water use if you feel that it is germane to the subject."

In Oregon and Washington, especially Oregon, uses of water have not generally been a matter of partisan politics with one exception, but that one exception has made it difficult to carry on factual, comprehensive development programs having in mind all beneficial uses of water.

In the squeeze between public power (Democrats) and private power (Republicans) orderly development and utilization of our water has been impaired. Hydro power being the source of the substantial amount of revenue from impoundments, it follows that other uses tend to be subservient to hydro power and at the mercy of the clash of power ideologies.

For some time I have been saying that, "It's time we forget the feud of public versus private power, or Democrats versus Republicans, and proceed with the development of our water resources." Such talk has cost me the active opposition of the public power devotees.

It's rather silly too, for I have never actively opposed the construction of a public power dam except Nez Perce, but have most violently opposed some private power projects.

Let us do a kindergarten study of water, just to have a look at basic values.

Nature has a cycle for water. Solar rays pick up the water from the ocean, prevailing winds carry it inland where ridges of the mountain areas push cool air currents upward, condensing the vapor which falls to the land in rain and snow. Moving laterally it flows across the land back to the ocean—thus the endless cycle; ocean to sky, to land, to ocean.

Man finds this cycle serves his needs in many ways which we call beneficial uses.

Many beneficial uses are possible from nature's cycle, but many uses, to be fully available, are only attainable by some delay in nature's cycle in the form of impoundments.

As I prepared this talk I was startled to discover that just we ordinary people are beneficiaries of nearly all of the beneficial uses of water.

Using myself as an example, I get *domestic* water from a well at our La Center, Wash., farm.

Our family-owned apartments are provided with *municipal* water from the Bull Run supply of Portland.

Our farm pond, S.C.S. designed, provides *irrigation* water, and a tributary of the Columbia furnishes our *livestock* with water.

Our 350 tenants are supplied *hydro power* generated in part by public power and distributed by private power.

For *pollution abatement,* we have the Willamette River to dilute Portland's sewage to which our tenants contribute.

My clients, Metropolitan Printing Co., print the Portland Telephone Directory, using 1200 tons of paper from Crown Zellerback Co. at Oregon City. This *industrial* use of water helps to pay my fee.

For *recreation* we operate our boat on the Columbia, taking *fish life,* and *navigate* the channel up through the locks of Bonneville and The Dalles.

Wildlife in the form of ducks and geese flying up from resting areas of the Columbia have provided me with hours of hunting.

There is only one more well recognized use of water, *mining,* with which I do not have a personal interest.

Now, what I am hoping you recognize is that all of these uses,

Domestic	Industrial
Municipal	Mining
Irrigation	Recreation
Power development	Wildlife
Pollution abatement	Fish life
	Navigation

are everybody's interest.

While our laws of the Northwest state that the water belongs to the public for the use of the people of the state, we have, in effect, proceeded to reduce water to private ownership.

Anyone desiring to use the water, if for a beneficial use, has traditionally been permitted to take such water as he needs and as he finds it available.

Our early day mining and agricultural economy grabbed off most of the natural flow of the streams of the Northwest, forcing those who came later, especially municipalities and industries, to store flood waters to make available a year-round supply.

Such a procedure has not provided for leaving an adequate flow in the streams to provide water for pollution abatement, recreation, fish life, and wildlife.

This situation has been a serious concern of fish and wildlife people. During low flows of summer, some streams have been pumped entirely dry, and others have had such a low flow that they could not absorb the wastes of civilization and support fish life.

Such conditions frequently have stirred the public to indignation meetings directed at cities and industries which were dumping wastes into the streams.

This indignation has almost never been directed against the consumptive user who pumps the stream so low as to make it incapable of carrying away effluents, or against those who have destroyed or limited the capacity of the watershed to retain water.

Fish interests have long urged that a minimum flow, or what might better be called a "desired base flow," be established for all streams, but they have had very little public support for such a program.

Actually, industry has a stake in such flows and should be the one to take the lead in their establishment, but I have never attended a meeting—State Water Board or others—where industry made any effort on behalf of such flows.

I have never known of industry in the Northwest giving any attention to the condition of the watershed that provides the water.

It is a well recognized fact with "resource" people that good land management can substantially reduce flood water. That is, properly vegetated land whether it be grass, farm crops, or trees, provides an *in* soak that will slowly find its way back to the stream and do much to firm up summer flows. In general, good land use, just to prevent erosion, requires that the lateral movement of water be delayed—certainly not accelerated.

Now I dislike to rub your professional noses in this situation by repetitious words, but the fact that you have not as an industry, busied yourselves with problems of minimum flows of streams and land management, indicated the necessity.

You who are connected with the industry located on the Willamette River, know the limiting factor of the effluents from your mills the river will absorb is determined by the flow of the stream. Add another 2000 c.f.s. and your trouble with the State Sanitary Authority would be lessened.*

Prior to the creation of the Oregon Water Resources Board in 1955, there was no way to legally establish a minimum flow for a stream. Now it can be done by the order of the Board and has been done on the Umpqua River. A like order is being announced for the Rogue River.

Thanks to the Army Engineers, a minimum flow—much

* Read: *Improvement in Pulp Mill Effluent Control on the Willamette River During the Summer of 1958,* by Dr. Isaiah Gellman and Eben L. Owens.

too low—has been maintained in the Willamette River by releasing stored water.

My research as to the situation in Washington and other Northwest states has not been completed to a point where I can report to you the law or procedure in these states, but it appears that there is no Board or Commission having the necessary authority.

You have a common cause along with recreational, fish, and wildlife people. All of you must have flowing streams.

Live flowing streams can be had by (1) good management of the land of the watershed, (2) by impoundments, and (3) limitation on diversions for consumptive uses.

Inasmuch as normal flow of streams has, in most cases, been reduced beyond safe limits by legal appropriations, a desired base flow can in such cases only be secured by storage of flood waters to be released as the flow requires.

Such storage can often be used as flood control and for hydro power, which will sometimes provide the necessary financing, but generally increasing the flow of the stream has not been a benefit which has been persuasive on Congress or state legislatures, or those individual agencies who develop the impoundments.

This desired base flow is a public benefit. Considering that all water belongs to the public, people often ask why not just fix the flow and not allow any diversions when the streams fall below that point?

The answer is quite simple, but still not understood by many. By our Western water law the public water becomes private property when appropriated for a beneficial use and cannot be taken without the exercise of eminent domain—that is, a taking under authority of law with adequate consideration.

It's possible that I have confused you with this talk of minimum flows and desired base flow. If you are confused you are not alone, for the engineers who do planning are having difficulty reducing either of these terms to a mathematical formula.

There is no exact formula, and no one agrees as to so-called criteria for determining such flows.

To me, it is the amount of flow of water necessary to keep a stream alive. Some authority must fix the flow in cubic feet per second and deny the right to further appropriate the water when the flow drops below that point.

Now some of you in the pulp and paper industry are going to say, "Why should we concern ourselves with this minimum flow of the streams?"

In none of your most optimistic reports have I found any prediction that you are going to purify the effluent of your plants to drinking-quality water. In fact, you will do well to provide a discharge in which fish could survive. This being true, you just have to have a flow of water in volume sufficient to absorb this discharge.

By now you have the viewpoint of this conservationist. Water, like other resources, is for the service and use of the people.

There is no exact formula or equation for multiple use. Of necessity our concept of multiple use must change and adapt itself to the needs of people.

The multiple use of 1900 is not the multiple use of today, and certainly it will not be the use of tomorrow.

The words, *multiple use*, indicate a sharing, a pro-rating, a division. It follows that such use brings conflicts and compromises.

How about the rights of those who give up a use of water that some other user may profit?

In the field in which you are most interested, we get around the situation nicely by the legal concept that pollution of water has never been a right—at the best it was and still is, only a privilege.

You who rank some seniority will remember the privies that once hung suspended over the water along the banks of our streams. They were replaced by a new use of water—the water closet, but the sewage was still dumped into the stream through a pipe and was now accompanied by tons of tissue instead of sheets from the mail order catalog. Demands of the use of the waters for other purposes have long since stopped this privilege just as it has stopped you from dumping untreated effluent.

For every privy that once lined the banks of our streams, you can now find a houseboat or some other type of river craft, including thousands of sports crafts, dumping human excrement directly into our waters. Is this a right for the use of water that goes with navigation?

We who call ourselves "conservationists," and you who call yourselves "industrialists" have a common cause. Your survival and the welfare of the people we both serve depends on the wise use of our natural resources, the first importance of which is water.

Presented at the joint meeting of the Pacific Section of TAPPI and the Pacific Div. of APPMSA held at Gearhart, Ore., May 21–23, 1959.

Bio-Assay Techniques Used in Tests at Simpson Paper Co.

D. J. Clark

In the fall of 1957, The Academy of Natural Science, Department of Limnology, at Philadelphia made a series of bio-assay tests on the wastes of Simpson Paper Company at Everett, Wash. The purpose of this project was to determine the acute toxicity of these wastes on the fish which migrate up and down the Snohomish River. As the only fingerlings available at this time were silver salmon, these were used for the tests. We felt that a description of techniques could best be made by explaining the procedures, computations, results, and conclusions of the experimental work on these wastes.

Before discussing this experimental work, I should like to briefly describe our operation at Everett, Wash. Ours is an integrated kraft pulp mill producing some 100 tons of bleached pulp daily, and a three-machine paper mill producing 125 tons of printing and writing papers. We also have a deinking plant which processes about 40 tons of slick-type magazines daily.

Although a kraft mill burns a major portion of the pulp mill liquors to recover the active chemicals, there are, however, a portion of the chemicals which are not recoverable. In a deinking operation, a relatively large percentage of the weight of magazines is lost in recovering the usable fiber. This consists of mineral pigments, emulsified ink and fines. In any papermaking operation there are certain soluble chemicals, pigment fillers, and fiber which are lost, even though every effort is made to keep these losses to a minimum. The effluents from these three departments, containing the wastes, are combined into a single effluent before being discharged into the river. It was representative samples of this combined effluent which were used for the acute toxicity tests. Three separate samples were collected, each over a period of 24 hr. and considered to be representative of the normal wastes discharged.

The fingerling silver salmon for this experimental work were secured from the state hatchery at Skyhomish through the courtesy of the Department of Fisheries, and transported to the mill in barrels. These fish were approximately 10 to 12 cm. long and 10 months old. They were acclimated to the dilution water for a few days in a holding trough through which water from the river was pumped directly and continuously. Each fish was used for only one test and only fish which appeared to be in good health were used. No food was supplied the fish during the actual tests periods, but the stock fish were fed daily.

The experiments were carried out in 5-gal. glass jars, each equipped with an air dispenser as a source of oxygen, and to

D. J. Clark, Simpson Paper Co., Everett, Wash.

keep the test solution well mixed. The experiments were conducted in an unheated portion of the mill where the temperature remained fairly constant. Records of pH, temperature, and dissolved oxygen content were determined for each test solution at least once every 24 hr. The range of operating conditions were as follows:

Temperature	11–15°C.
pH	6–8–7.4
Dissolved oxygen	6–12 p.p.m.
Dilution water	Pumped directly from the river through the fire line. This water was flowing through holding trough constantly, and water for tests was secured from the same line.

The procedure for starting the experiments was that the amount of waste necessary to give the correct concentration was added to the dilution water in the 5-gal. jars and thoroughly agitated. For instance, for the 75% concentration, three parts of waste and one part dilution water was used. The pH, temperature, and dissolved oxygen content tests were made, and as soon as test conditions were found to be within the desired ranges, five fish were introduced to each jar and the air dispenser started. A control test jar was maintained with each series of tests. All conditions were maintained the same for the controls as for the test jars, except that 100% river water was used in the controls. No deaths or impairment of fish was detected in any of the controls. As you can see in the following tables, several series of tests were made on the solutions in each test jar during the first 24 hr., and then once for each additional 24 hr. of the test period. Also included in this record is the per cent survival of the fish.

These tests are carried out in keeping with the recommendations of the Committee on Research, Subcommittee on Toxicity, Sec. III, Federation of Sewage and Industrial Waste Association published in 1951 by Doudoroff, and by Hart, Doudoroff, and Greenbank in 1945. These tests are designed to determine the concentration of toxic materials which would kill 50% of the exposed organisms in a given interval of time. This is expressed as the median tolerance limit (TL_M). The time intervals are 24, 48, and sometimes 96 hr. On these particular series, the test periods were extended to 144 and 168 hr. The concentration of chemical or waste is expressed in parts per million, or in per cent by volume. In these experiments the dilution is expressed in per cent by volume. If exactly 50% kill is not experienced, but kills above and below the 50% is found, the TL_M can be found by plotting the points on semilog paper.

From the $TL\Delta$, the biologically safe concentration is calculated by a formula given here, and in essence means that a factor of 0.3 is used to convert the TL_M to biologically safe concentration.

$$BCS = \frac{(48 \text{ hr } TLM)\ (0.3)}{S^2} \qquad S = \frac{\text{24-hr. } TL_M}{\text{48-hr. } TL_M}$$

This method of calculation is also suggested in the publications by Hart, Doudoroff, and Greenbank.

Table I

Median tolerance limits: 24-hr. TL_M, 81% dilution by volume; 48-hr. TL_M, 81% dilution by volume

Concentration of waste, % by volume	*Total exposure, hr.*	*Per cent survival at cond. of exp.*
100	12	0
95	24	0
87	120	20
87	72	0
75	120	100
75	120	40
65	144	100
65	120	100
56	144	100
56	144	100
42	144	100
42	144	100
32	168	100
32	144	100
24	144	100
24	144	100
10	144	100
10	144	100
1	144	100
Control	168	100
Control	144	100
Control	48	100

Biologically safe concentration: 24.3% dilution by volume.

Table II

Time, hr.	*Per cent survival*	*pH*	*D.O.*	*Temp., °C*	*Per cent survival*	*pH*	*D.O.*	*Temp. °C.*
	100% Waste (Sample 1)				95% Waste (Sample 3)			
0	..	7.4	7.6	13	..	7.4	8.6	11
6¼	80	7.6	7.6	..	..	..	..	..
12	..	..	..	..	0	7.6	10.0	11
14¾	0	7.6	7.6	15	..	..	..	..
	87% Waste (Sample 2)				87% Waste (Sample 3)			
0	..	7.8	6.8	14	..	7.4	6.2	11
7	80	7.8	8.8	13	..	..	..	..
11	40	..	..	..	..	..	..	..
24	40	7.6	7.8	14	20	7.6	10.6	11
48	40	7.4	8.8	11	20	7.6	10.0	12
72	20	7.4	10.2	11	0	7.6	9.6	12
96	20	7.4	8.6	12	..	..	..	..
120	20	7.4	10.2	12	..	..	..	..
	75% (Sample 1)				75% (Sample 2)			
0	..	7.6	7.4	15	..	7.6	8.0	14
12	..	..	..	..	40	7.6	8.2	..
24	100	7.4	8.6	14	40	7.6	8.8	14
48	100	7.6	9.2	14	40	7.4	8.8	12
72	100	7.6	8.8	11	40	7.2	6.6	11
96	100	7.8	10.0	11	40	7.8	9.6	12
120	100	7.8	9.8	12	40	7.6	8.8	12

In Table I the results of acute toxicity experiments are listed. It is seen that when the fingerling salmon were subjected to 100% and 95% mill wastes, the per cent survived was zero. At 87% concentration there was also zero survival after 72 hr. with waste sample III, and 20% survival after 120 hr., with waste sample II. At 75% concentration there was 100% survival after 120 hr. with waste sample I, and 40% survival after 120 hr. with waste sample II. The next lower concentration was 65% and there was 100% survival with waste sample I and II as well as with all lower concentrations. The tests were continued down to 10% dilution.

Table II presents the same information with additional detail covering the experiments where fatalities occurred. This gives the per cent survival at various time intervals as well as the routine tests made of the test solutions periodically during the experiments. Table III gives the same information for the controls. It is seen that there was 100% survival on all the controls over the duration of the tests.

The conclusions from this experimental work as reported to us by the Academy of Natural Science was, and we quote, "The biologically safe concentration for the acute exposure of silver salmon to the Simpson Paper Co. plant waste would be 24.3% dilution by volume. Since each critical concentration was run in duplicate, the average of results for a given concentration was used in plotting points on the semilogarithmic paper. For example at 87% in 24 hr., there was 20% survival in one test jar and 40% in the other, thus giving an average of 30%. Due to the small number of fish used in each individual tests such as variation at critical concentrations is not unusual."

We consider the results of these tests quite satisfactory. The biologically safe concentration of 24.3% is interpreted to mean that silver salmon fingerlings would be expected to survive when migrating past the mill, providing the con-

Table III

Time, hr.	Per cent survival	pH	O.D.	Temp., °C.
		Control 1		
0	..	7.0	10.6	13
24	100	7.2	10.4	15
48	100	7.2	10.0	14
72	100	7.2	10.2	14
96	100	7.2	10.6	11
120	100	7.2	10.6	11
144	100	7.2	10.8	12
168	100	7.2	10.9	12
		Control 2		
0	..	7.4	10.2	15
24	100	7.2	9.8	14
48	100	6.8	8.0	14
72	100	7.2	9.8	12
96	100	7.2	10.0	11
120	100	7.0	10.2	13
144	100	7.1	10.2	12
		Control 3		
0	..	7.2	10.1	11
24	100	7.2	9.8	12
48	100	7.0	9.6	12

centration of mill wastes in the river did not exceed this 24.3%. We have calculated that at minimum river flow this concentration would be less than 2%, once the mill effluents are dispersed into the river flow. Within the past year, a dispersing line has been put into operation at our outfall which carries our combined effluents into the deep river channel. With this addition, we are of the opinion that there is rapid dispersion of our wastes in the river, which insures that the concentration of wastes is considerably below the biologically safe limit.

As the dissolved oxygen concentration was maintained at better than 5 p.p.m., any harmful effect due to oxygen demand of the wastes was not determined in this experimental work. We have determined, however, during our summer and fall monitoring programs that the discharge of our wastes into the river reduced the dissolved oxygen content very negligibly. During these critical months we have sampled the river at eight different stations ranging from two miles upstream from the mill to a point about three miles downstream. Chemical tests indicate that there is very little reduction in dissolved oxygen content as a result of the discharge of our wastes.

Presented at the joint meeting of the Pacific Section of TAPPI and the Pacific Coast Div. of APPMSA held in Gearhard, Ore., May 21–23, 1959.

Bio-Assays—A Tool of Industry

Norman S. Lea

We of Scott Paper Company at Everett, Wash., firmly believe that our dispersed spent sulphite liquor in the concentrations occurring in Port Gardner at Everett is not harmful to salmonoid fish nor to the food chain upon which these fish live. Because this is a positive statement, I am sure you will be interested in hearing some sound scientific evidence.

A large part of this proof lies in the field of bio-assays. What is a bio-assay? A general definition is: the estimation of the strength of a variable in terms of its effect on a living organism. In our particular case, the variable is spent sulphite liquor, and the living organisms are represented by Pacific salmon, shrimp, and diatoms.

To carry out a bio-assay effectively, the standard method of controls and variables is used. The control is the living organism—fish, shrimp, or diatom—living in complete health in an aquarium. The variable aquaria are then set up with conditions as close as possible to the control, the only difference being the introduction of varying amounts of spent sulphite liquor. By observing the results on the organism at the various levels of spent sulphite liquor and comparing with the like organism in the control tank, it is possible to obtain the mortality threshold index, or, that concentration of spent sulphite liquor required to bring about a 5% mortality among the test organism in the variable tanks in 30 days' time.

Norman S. Lea, Technical Control Manager—Pulp, West Coast Div., Scott Paper Co., Everett, Wash.

We have in our files at Everett the results of thousands of monitoring chemical tests which we have conducted over the years in the harbor area of Everett and in the Snohomish River. These results dramatically illustrate the effectiveness of our underwater pipeline in dispersing the spent sulphite liquor and blowpit washings from the two sulphite mills in the area. This pipeline is jointly owned by the Scott Paper Co. and the Weyerhaeuser Timber Co. The dispersion, at a depth of 340 ft. on the harbor floor and through 110 stainless steel nozzles on the last 1000 ft. of the 3000 ft. long pipeline, results in an average spent sulphite liquor concentration of 45 p.p.m. at 10, 50, and 100-ft. depths in an area bounded by an arc of 1800 yd. radius centered by the diffuser line surge tank.

We have long realized that this 45 p.p.m. level of spent sulphite liquor is a very low concentration, but chemical tests such as this do not tell the complete story, and we could not be sure about the toxic effects at this concentration until substantiating bio-assay work had been conducted on the marine biota. Certainly, past lack of knowledge in this field has placed any plant using a portion of public waters for waste assimilation at a distinct disadvantage. It is here that the bio-assay has been of immense value to industry and has really come into its own.

In 1952 the Washington State Department of Fisheries and the Pollution Control Commission carried out an extensive bio-assay on the Chinook, the pink and the silver salmon, to determine the effect of spent sulphite liquor on these organisms. In their Research Bulletin No. 1, entitled: "Toxic Effect of Sulphite Waste Liquor on Young Salmon," they presented the following mortality threshold indices:

	Days old	10% SSL, p.p.m.
Chinook salmon	38	1175
	280	600
	305	560
Pink salmon	102	1550
	198	530
Silver salmon	351	1230
	412	1015

In 1958 the Academy of Natural Sciences in Philadelphia conducted bio-assays for the Northwest Pulp and Paper Association. The purpose was to obtain toxicity levels on organisms, including those representing important steps in the food chain. Therefore diatoms, shrimp, and juvenile silver salmon were selected for the studies. They found the safe biological limits to be as follows:

	10% SSL, p.p.m.
Silver salmon	1096
Shrimp	7320
Diatoms	1460

These results are in line with those of the Department of Fisheries. Also, recent work would indicate that had aged spent sulphite liquor instead of fresh been used, these values would have been higher, due to the breakdown and assimilation of spent sulphite liquor with time.

The question is raised whether spent sulphite liquor is affecting the food chain. To answer this question, the Fisheries Department conducted field biological assays in

1954 and 1955 in Port Gardner and Skagit Bay. The control here was Skagit Bay, which is an area not influenced by waste discharge, and the variable was Port Gardner, which has high waste discharge. The results are reported in "A Biological Assay of Pollution in Port Gardner in 1954 and 1955," by J. E. Lasater. Special attention was paid to the study of copepods, for, without exception, all marine fish feed on copepods in some part of their life cycle. Some of the conclusions are as follows—I quote:

> 1. The number of species of copepods found was basically similar in Port Gardner and Skagit Bay in 1954, but peaks occurred at different times.
> 2. The number of copepods per 1000 l. in Port Gardner was lowest in the area of heavy pollution along the Everett waterfront, but was about the same as the numbers found at comparable salinities in Skagit Bay in 1954.
> 3. Stomach analyses of young salmon showed nearly all were full and no food shortage was indicated.

Another point I would like to make is that many leading biologists are now suggesting fertilizing properties for diluted spent sulphite liquor. Willis M. Van Horn of the Biology Group at The Institute of Paper Chemistry, made the following statement in this connection in 1953:

> I have observed that as long as adequate oxygen resources are available in a stream, the sugars in sulphite waste liquor will stimulate the growth of bacteria and of fish food organisms.

This may well be the case at Everett.

I hope that the information presented today clearly shows the value of the bio-assay to industry. In our particular case, before its application to spent sulphite liquor, we knew from chemical tests that our underwater dispersal line had reduced the spent sulphite liquor concentration to a low value, but we could not be sure of the improvement on the marine biota. Here was where the bio-assay came into its own as a valuable tool. Through the use of the bio-assay, we were able to scientifically apply our vast amount of monitoring data and to correctly evaluate the effectiveness of our deep water dispersal line.

Yes, gentlemen, the bio-assay is here to stay. Its correct and objective use can contribute greatly to the recognition of responsible industry as a solid member of the community, dedicated to the protection of the multiple use of one of our greatest natural assets—water.

Presented at the joint meeting of the Pacific Coast Div. of the American Pulp and Paper Mill Superintendents Association and the Pacific Section of the Technical Association of the Pulp and Paper Industry, held in Gearhart, Ore., May 22, 1959.

Fisheries Resources and Landings of the Northwest and Their Relations to Wastes

Roger Tollefson

In the course of preparation of this discussion I was tempted to re-title the discussion "A Problem in Perspective," since this is what any discussion of fisheries landings versus water usage becomes.

Fisheries stocks, and especially their landing statistics, as used in conjunction with industrial problems are primarily the results of many factors, rather than any sort of causative factor of their own. Therefore, in this general problem, we must always be careful to consider these stocks or statistics in relations to the many factors operating on them—a problem in perspective. Let us look first at the over-all picture of fisheries resources as defined by their commercial production or landings.

The United States fishing industry, including Alaska, lands of the order of between 4 and 5 billion lb. of fish per year, valued at the order of one third of a billion dollars value to the fishermen. The Pacific Northwest (here being used as including Washington and Oregon) is comparatively low in poundage of fish landed as compared to grand total or to many other areas; normally landing a total of about 200 million lb. of fish per year (or about 4% of the United States total); but rates comparatively high in dollar valuation, the landings being worth about 30 million dollars per year (or about 8% of the United States total). This is primarily due to the high per pound valuation of our salmon fishery, which dollar valuation is equalled or exceeded only by the Gulf Coast shrimp industry, the Pacific tuna fishery, and the Chesapeake Bay oyster industry.

The importance of such an industry is obvious, but its importance differs, depending upon what group or situation is involved: again a matter of perspective. Its economic value to the people in the fishing industry and to the economy of the United States as a whole, is obvious; but I do not believe that industry should gage its importance by these economic standards. In the first place, should one be tempted to assess the comparative importance of fisheries and any respective industry in dollars and cents, let me comment that it is rare, if ever, that such a weighing of dollar values ever has, will, or should, finally resolve and problem or conflict. Industry should rather gage the value or importance of fisheries as related to themselves in terms of the sociological factors such as the public uses of the resources (such as sports fishing) the simple esthetic values of water usage, and the degree of organization and ability to obtain action on the public level by any respective fishery group. This last point especially by its very nature is frequently the best developed and most difficult to combat in the case of some of the smaller fisheries, thus making some of the smallest (poundage or dollar-wise) fisheries the most important in industrial problems. This sort of thing has been very strikingly demonstrated for example, in the State of Washington; where, for the past few years the comparatively small oyster industry, landing of the order of 10 million lb. of production valued at $1^1/_2$ to 2 million dollars per year (about 10% of the state's total), has probably focused more attention and stirred up more controversy in the field of industrial waste disposal than all of the remaining 90% of the state's fisheries have accomplished in the state's history.

What then are the values of fisheries statistics or landings? Despite my opinions expressed before, I will be among the first to argue for their use, and as to their value. They are a tool which at times will be used against industry but which in turn can and must be used by and for industry. These must be directed not just in a defensive line, but for purposes of true constructive analysis of situations, and for the resolving of these situations.

The simple and long established method of application of such data is to plot the landings of a given fishery over a period of years to find what trend, if any, is involved. If this trend should be downward in the presence of an industrial operation, then that industry frequently becomes the target for desired action. On the other hand to exaggerate for the sake of illustration, one could probably also plot the trend of some of our salmon fisheries and obtain a correlation with the number of telephones installed in the area, or the gasoline tax paid; and find equally true that the fish had been either talked to death, or hit at a crosswalk, as the cause of their decline. We must analyze and weigh much more than just the simple trends because of the danger of working with an incomplete picture, and the further danger of coincidental effects (regardless of degree of correlation) even when a fairly complete picture is utilized.

As an extreme but comparatively noncontroversial illustration of the danger of an incomplete picture in simple trend analysis, I would cite the situation resulting in the chinook salmon run in the Columbia River following the 1935 closure of the State of Washington waters to the use of fixed gear. If we compare the Washington catch on the Columbia for the 7 years immediately preceding this closure with the seven years just following it, we find that the chinook

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salmon landings dropped by 19½ million lb. or a percentage drop of about 39%. Utilization of these data alone could conceivably lead to the conclusion that the fish traps, etc., had been helping to maintain the chinook run in the Columbia. When, however, we examine the Oregon landings in addition to those for Washington, we find that these catches increased by over 12 million lb. and resulted in a combined value of only a 6% decrease, a figure entirely in keeping with the over-all trend existing through the period. Hence the removal of fixed gear in Washington had no appreciable effect upon the chinook salmon run; quite a different conclusion than that reached by an analysis of only part of the data.

As an example of a similar problem of the immediate present, let us consider the interpretation that should be placed upon landing statistics of either chinook or silver salmon in Oregon and/or Washington. Whereas most of the public appreciates the fact that the pink, the red, and the chum salmon runs have their greatest and deciding importance in the northern waters of especially British Columbia and Alaska, not nearly so many appreciate that in the case of chinook salmon for the 10 years from 1948 through 1957, Oregon and Washington combined, still amounted to only 35% of the Pacific total; and that in the case of silver salmon these combined catches were only 24% of the Pacific total. In light of the considerable data being obtained by biological investigators as to the intermingling of these stocks and fisheries thereon, and the extreme increase in proportion of ocean troll catch to total catch, we must be careful in assessing the results of activities within any given locality upon its respective run of fish since it is obvious that its assigned commercial catch represents only a portion of the total fishery operating upon that run.

As an example of a slightly different type of problem involving incomplete pictures, let us consider the salmon runs in the Columbia River. For any given species we find we will have a certain trend of catch of fish, evidencing the condition of the stocks. This value, however, merely represents the condition of the total of all the races or runs of that species that occur within the Columbia. Without a breakdown, at least into time and zone bases, coupled with true biological data, it would be possible to be overprotecting a given race or run while another might be heading toward comparative extinction. This same general situation has also been demonstrated in the case of the Fraser River watershed where it has been found that fish passage facilities must be maintained over an extended time period, rather than merely passing an adequate total number of fish at one time, in order to prevent shorting individual runs into certain of the rivers. This again emphasizes that we must consider any landing statistics in relation to as many of the related factors as possible.

Another extreme example of the condition of the fisheries resources was evidenced by simple commercial landings is illustrated in the State of California landings as influenced by conditions associated with the last war. When one compares the 1937 to 1940 landings with those of 1941 through 1944, one finds that the yellow fin tuna catch decreased by 43%; due, of course, to the boat restrictions in the southern off-shore waters. However, the over-all catch for the entire state was virtually identical; the difference being made up by a 5% increase in the sardine catch. An associated factor; namely, a price setting by the O. P. A. at a low level is attributed as the cause of the rock fish landings being cut in half—an economic factor resulting in an otherwise apparent decline in a fishery. As a still further factor, and an interaction of the foregoing situation, the switch in fishing intensity helped result in the introduction of the then new light balloon net gear which upon next being applied to the rock fish fishery then brought the landings back up to double what the original level had been.

It is granted that all of these examples listed are extreme and obvious that being why they were selected as illustrations; but surely no one can deny but that less obvious factors of this same type are continually operating upon our fisheries and must be properly assessed to correctly utilize the landing statistics.

Two specialized fields of application of landing statistics are in the direction of yield per unit effort values, and in determinations of comparative size and trends in size of individual fish comprising a run. It is granted that both of these applications are frequently very difficult to obtain with any degree of accuracy, but can be of considerable assistance when they are determined.

Another direction in which the use of these statistics is becoming of more and more importance, and should be even more intensified, is in the field of economic analysis. The fish industry is a business effected by more than just someone's desire to catch a fish. It is also plagued with all of the economic interactions and ramifications that beset any business. To a certain extent, this will even hold true of the sports fishery since even the most rabid sportsmen may occasionally have to reconcile his desires with his pocketbook. These landing statistics then become a basic tool in the detailed analysis of these economic factors.

An example of the application of such data to immediate practical problems of industry has to do with the timing of occurrence of certain seasonal fisheries in any given waters. The use of such data is especially obvious in the case of any industry that might be considering, or actually engaged in, any impoundment of any type of waste.

With the hope of possibly clarifying some of the problems involved in the field, let us very briefly go into the history of the development of this field. In approximately 1890 (varying with states) the various governmental agencies charged with fisheries management in the Northwest, began to publish annual or intermittent reports. These reports were, of course, interspersed with many very good scientific type observations and recommendations. As the organizations developed, the constructive quality of the reports also developed and in the course of these, various statistics were compiled and presented. As a result of state licensing of fishing activities it also became necessary to report on such activities. The big impetus in the reporting of commercial landings came when the various fisheries became taxed on a poundage basis. From then on the reliability of these reports gradually increased as the various states gradually tightened their taxing structure so that in the 1920's these collection systems had become quite highly effective. A major step in the case of Oregon was the inception of the procedure of making dealers rather than fishermen, basically responsible for reports and taxes in 1923; while a noteworthy step in the case of Washington was passage of Initiative 77 in 1935 which among other things, defined the boundaries of Puget Sound and gave rise to a system of more accurately defined catches. A constructive step toward increasing the availability and usability of statistical data occurred with the formation of the Pacific Marine Fisheries Commission (the Tri-State Compact) in 1948 when for the first time all three Pacific Coast states were able to get together and begin cooperative and more comparable collection of these vital statistics.

Another major step in both Oregon and Washington has been in the physical processing of reports and statistics when first Oregon, and later Washington, in the middle 1940's and 1950's turned to the use of IBM processing of their data. This for the first time permitted exact and detailed breakdowns and compilations of their data, still within the realms of economic practicability.

The original or basic impetus to such data has, of course, been that of taxes. However, there has been a trend in especially the past ten of 15 years toward accumulating such data for biological and administrative purposes as well as the collection of taxes. This trend is fortunately still con-

tinuing at an ever increasing rate. However, I contend that we should continue to change in concept to the point that the primary purpose of gathering of statistical data be made for the utilization of biological and administrative purposes.

In the course of development of these procedures, many points of procedures were followed that now give us considerable trouble. It is understandable that such situations would occur in the development of any such major program and we certainly should not berate the pioneer activities. Nonetheless, it is of importance to recognize the shortcomings and evaluate the resulting data in light of them. For example, some of the original data was gathered merely on the basis of voluntary questionnaires to the fishermen, frequently under conditions where there may have been a human tendency to slant or prejudice the answers for personal business reasons. This is also true of some of the work of some of the governmental agencies themselves, where again (especially in the cases of hatcheries) there may have been either a deliberate, or unintentional, falsification in essence of the data.

Because of the very physical nature of such gathering of the data and the conditions of the time, many desirable aspects were either neglected or impossible of obtaining. The absence of such data today sometimes has led to generalized conclusions that may or may not be correct. Some of these major commissions continued until as late as 1948 to 1951 and of course, even today there are certain omissions that are continuing. Frequently as a matter of necessity, certain important data even today is being gathered on the basis of partial counts, sampling or estimates. We can appreciate the problems in this respect and recognize the unavoidability of many of these undesirable situations, however, we must still be sure we are aware of them.

I would summarize the needs of the field of fisheries statistics under three general headings.

1. We must ultimately place the gathering of such statistics under the complete control of the research and administrative groups with the primary purpose of the statistics being for their utilization and with any tax purposes being definitely secondary.

2. The field must be expanded to permit thorough and complete analysis of all problems. Granted that perfection will never be obtained in this respect, there is still considerable room for improvement before we need to worry about striving for the impossible.

3. Since no single agency could ever collect all the data required for all geographic areas and since many of our fisheries must be dealt with on an over-all basis, it is important that all agencies collecting such data, adopt and conform to uniform practices of collection and presentation so as to permit their later integration where required.

Industry also has a responsibility in this field. All affected industry, not just the fishing groups, must verify the desirability of such material and support both practically and politically, those people charged with the collection of such. There is frequently a tendency in some groups to feel that certain such activities may be directed against them; however, this may frequently be merely the result of misunderstanding, a matter in which neither faction is blameless, and one which never can be solved by merely saying, "You first."

Industries with waste considerations also have an obligation to themselves to become better acquainted with this type of material, its limitations, and its uses for there can be no question but that the material does have a definite potential as a tool leading to the best ultimate answer. Industry, anticipating the possible occurrence of a conflict or a question, either from expansion, development or what have you, also has an obligation to the regulatory agencies involved: to work with them in advance of any possible problems as far as it is possible, since these agencies must know and appreciate the industrial problems in order to best serve and assist all groups involved in water usage.

No one expects that all individuals or all groups will ever see eye to eye on any problem as complex as that of water usage, but I believe that a sincere effort by all groups to examine such matters as fisheries statistics in their complete perspective, rather than solely from a selective favorable aspect, will go far toward constructive settlement of many of the conflicts.

Presented at the Ninth Pacific Northwest Industrial Waste Conference, Seattle, Wash., April 2–3, 1959.

Accomplishments of Mills on Water Quality Control

S. Kepple Pratt

The Northwest pulp and paper industry is this area's third largest business. It is the second largest industry dependent upon the area's natural resources, where two thirds of the western watershed and one tenth of the nation's timberlands are concentrated. Proper management will afford a continuous supply of these two resources not only for present needs but to sustain future expansion.

This can and is being done without harm to the other industries and activities of the Pacific Northwest. As a matter of fact, the growth of the pulp and paper industry tends to strengthen the position of the other industries in the world's markets.

First, let's look at the progress we've made in conserving our timber resources. A fine example is a comparison of pulpwood consumption from 1947 to 1958. In 1947, 3,412,-032 cords were used and in 1958, 5,736,424 cords were used. However, during this 11-year period, logs used for pulpwood decreased while the use of farmerwood and wood chips increased. This fact takes on added significance by virtue of the fact that the grade of logs now being taken out of the forests consists more and more of salvage and re-logged trees which were formerly wasted.

This fact brings into sharp focus the pulp mill's direct contribution to the economy of other industries of the state. Pulp mill dollars, earned through utilizing once wasted wood, supplement other components of forest industry including logging and lumber.

Even agriculture is benefited by the pulp industry, since the purchase of pulpwood from tree farms represents a major portion of the agricultural dollar.

The growth of the industry is in large part due to the availability of its raw material. In 1947, the West produced 2,046,432 tons of product or 17% of America's total production. In 1958, the West manufactured 4,163,529 tons representing 19% of America's total production. However, the South has expanded at a much greater rate than the West despite our more favorable supply of natural resources.

These figures only highlight the surface in showing the pulp mill's value to the economy of the Northwest. It must also be remembered that many allied industries—chemical companies, material handling companies, engineering firms—are located in the Northwest to service the pulp and paper industry.

The industry shoulders a tremendous, two-pronged responsibility—both to maintain its position and thus the economy of the Northwest, and secondly to conserve and protect the natural resources of timber and water upon which it sustains itself.

It has been pointed out that technological advancement has made it possible to utilize wood products to the extent that wood cut for pulp is decreasing while the wood actually used by the industry is increasing.

Let us take a look at the other natural resource—water. The industry has accomplished much toward the abatement of water and air pollution and is continually putting nonproductive dollars into a successful battle to further improve

S. Kepple Pratt, Superintendent of Manufacturing, St. Regis Paper Co., Tacoma, Wash.

conditions. These improvements continue to take place even while the industry is expanding rapidly.

The industry has continually sponsored research and development programs to add to the technical knowledge of recovering and using once wasted materials.

It is easy to conceive the fields of by-products are vast—but they are not always obvious. Recovery of some types of waste is a problem—sometimes not feasible. Dispersion of wastes to everyone's satisfaction is difficult. The problem of abatement of waste looms as a gigantic research problem, both basic and functional, since much is yet to be earned of the complete structure of waste and its effect.

In the 12-year period, 1947 through 1958, $2,982,967 was spent by individual companies in the Northwest for research, of which $827,777 last year was for research on air and water pollution.

In the same 12-year period, $1,098,731 was spent on contracts to outside firms and organizations, of which $216,872 was spent in 1958.

Steps are taken daily in response to favorable research. $10,498,579 was spent from 1947 through 1958 and $1,754,857 in 1958 for construction of facilities which had water pollution abatement as the direct objective. In the same period, $26,861,821 was spent—$3,080,496 of this in 1958—for construction of facilities which had water pollution as a corollary object.

But with all this expenditure, pulp and paper mills still must dissipate their wastes into the various bodies of receiving water. Every mill in the Northwest is attacking this problem with an intensity in direct relation to local conditions, and in most cases during the past few years research has been greatly intensified.

As an illustration, in 1947 $45,650 was spent in monitoring discharges and wastes. In 1958 over six times that amount—$312,176—was spent in monitoring. This does not include work on processes for better efficiency which results in better control.

To get a clearer picture of accomplishments in water and air pollution abatement, and I am including air because of its common problems, the achievements can be categorized under three headings: (1) direct, (2) corollary, and (3) incidental.

The category Incidental does not minimize the achievements made here, which are, in fact, among the most important in pollution abatement and control. Modernization and thought control are two subheadings under incidental.

Now, when equipment should be replaced or a process modernized, the decision of mill managers is increasingly influenced by the pollution abatement advantages of the new or modernized machinery. For example, a new washer line was recently installed at the St. Regis Paper Co. mill in Tacoma, Wash. Its prime purpose was not pollution control but the decision reached to replace the inefficient old line was accelerated by the chemical loss to the sewer which was both a matter of economics and pollution.

Most important of the incidental items is "thought control," in which we must impress upon our employees the importance of water quality control. It can be done by distributing literature and reports to them as well as pointing out accomplishments. Many mills use conductivity meters in low flow minor sewers to show ups and downs. In many cases, the charts do not show major pollutants but they certainly point out to the operators the minor contributories which make them water control conscious.

Direct and corollary categories approach the problem of pollution abatement more specifically. Direct items can be subheaded thus: (1) water sampling and survey, (2) control installations, (3) educational, and (4) research.

An excellent example of water sampling and survey work and its results is the Willamette River Survey.

This was an extensive survey to establish the amount of pollution and the self-purification capacity of the river.

Station 1 is located below Albany, Ore., and above Western Kraft Corp. The stations continue down the river, bracketing towns and mills until the river flows into the Columbia.

The survey showed marked improvement in quality of the Willamette River water last summer from effluent control programs of five pulp and paper mills along its banks. Control progress begun in 1950 shows steady improvements each year, with the last year the best to date. This was reported by the National Council for Stream Improvement, summarizing a cooperative stream survey program being conducted by the council, the mills and the Oregon State Sanitary Authority. Reporting at the Ninth Pacific Northwest Industrial Waste Conference, the council said improvements included:

1. A 36% decrease in the amount of organic waste discharged—the result of new equipment, better housekeeping, and increased impoundment during low-flow periods of the river. These improvements increased the ability of the river water to sustain life.
2. The "biological oxygen demand" (B.O.D.), a measure of the waste load, was reduced considerably below levels required by the state authority.
3. This resulted in an increase in dissolved oxygen—a beneficial effect on stream life.
4. Release of reservoir waters during low-flow periods contributed to these stream improvements.

The council said further study of the releasing of reservoir waters during low-flow periods should be made in planning further measures for improvements of the Willamette. Size of the cooperative industry program is indicated by the fact that the five mills alone took more than 2300 analytical tests, in addition to council and state tests made weekly along a 92-mile stretch of the river. Impoundment and barging downstream of pulp mill effluent, and general tightening of mill operations effected the changes. Cooperating in the long-range program along the main stem of Willamette River are Western Kraft, Oregon Pulp and Paper, Spaulding Pulp and Paper, Publisher's Paper, and Crown Zellerbach.

Surveys of virtually every body of water in any way connected with pulp mills are being carried on by individual mills or groups of mills to accomplish the same end. Such monitoring programs are extensive in the Grays Harbor area by Weyerhaeuser Timber Co. and Rayonier to measure influences of wastes and to aid in operating the vast storage lagoons which have been installed to protect the receiving waters. Rayonier installed recently a butterfly valve and controller to allow the mill to control the amount of effluent discharge at a fixed rate and to govern when the discharge takes place. Normally all strong liquors are stored between late April and October. During other months the discharge occurs during outgoing tides.

A somewhat similar installation is the system of lagoons at Weyerhaeuser Timber Co.'s mill at Cosmopolis. The lagoons retain the effluent and a gate controls the discharge on an outgoing tide.

This type of water control program and the Willamette River program typifies the action taken when surveys indicate such measures are necessary for high standards of water control.

Other control work of this type includes dispersant lines and sewer measuring devices. A Parshall flume measures the effluent from the St. Regis mill is an example.

Many surveys could be classified as research or educational since no immediate action is required. The more direct educational activities of the industry are the efforts of the Technical Committee of the Northwest Pulp and Paper Association. Under the auspices of the association, a number of biological seminars have been held to advance the technical knowledge of the industry in its efforts to recognize and control the effect of pollution on marine life. The following seminars have been held:

Dr. Ruth Patrick, Curator of Limnology, Academy of Natural Sciences, Philadelphia, "Fresh Water Biology."

Dr. Willis Van Horn, Chief of the Biology Group, The Institute of Paper Chemistry, Appleton, Wis., "Aquatic Biology and Surface Water Management."

Dr. Gordon Gunter, Director of the Gulf Coast Research Laboratory, "Puget Sound Oyster Problems."

Dr. E. M. Wood, Fisheries Pathologist Consultant, "Pathology, Histology, Physiology Relating to Fish Kills, Pollutants, and Industrial Waste."

The association's public relations committee also conducted a seminar designed to assist in methods of effectively publicizing accomplishments of the industry. Guest speaker was Arthur Van Vlissingen of the Pulp and Paper Information Service of Appleton, Wis.

Now under the category of direct pollution abatement methods, we have explored: (1) water sampling and survey (2) control installations, and (3) education. Let us look at the fourth subheading—research.

Research embodies many facets including surveys, basic research on elements, by-products research, and process research.

Surveys recently completed or in progress include "Fisheries Statistic Study," the "Economic Survey," "Accomplishment Survey," "Survey of Fiber Recovery Installations," and "Survey of Hydraulic Barker Effluent Screens."

"The Fisheries Statistic Study" is a survey of fishery statistics of the Northwest to further the knowledge of marine life by studying the past history and life patterns of the fish. With this and an over-all picture of the environment surrounding aquatic life, technicians of the pulp and paper industry can obtain a better understanding of the problems which we face. The survey has developed very well and much of the reliable data has been compiled and is now in the process of assimilation into a report.

The Economic Survey, compiled each year by the Northwest Pulp and Paper Association, was the basis for figures given earlier in this report. Other numerical data used in this paper was taken from the association's Accomplishment Survey.

The "Survey of Fiber Recovery Installation" and the "Survey of Hydraulic Barker Effluent Screens" are examples of mutual aid between mills. They indicated the relative efficiency of fiber and bark recovery correlated with the equipment used. This enables mills with a bark and fiber loss problem to evaluate equipment used at other mills and so to determine which types would best suit their own operation.

Many individual companies and mills are also carrying on their own research programs in marine laboratories, in river and water studies, by-products, etc. They jointly contribute invaluable information to the National Council of Stream Improvement, Sulphite Pulp Manufacturers' Research League, The Institute of Paper Chemistry, and the University of Washington, and other over-all organizations.

The research programs vary from basic element studies to pollution abatement work. An example of such a research program in the development state is a pilot plant (a miniature model, not for actual production) to test a process developed by the University of Washington scientists.

The pilot plant seeks to eliminate odorous components of gases from kraft pulp mills. It is in initial operation at the St. Regis mill in Tacoma. Developed through the University's Pulp Mills Research Project, the process came out of scientific research conducted during the past 10 years. St. Regis was chosen as the test site because of its proximity to the University. This will expedite supervision and scientific control by University researchers.

The mill, a member of the Northwest Pulp and Paper Association which sponsors the research, contributed construction and operation personnel. Findings, of course, will be made available to NWPPA members. The work could take a year, although preliminary results may be available sooner. Although researchers are optimistic, they caution that many a laboratory scale process did not result in success in either the pilot or full-scale plant.

The process, scientists hope, will collect and concentrate gases from several sources, and will dispose of them by burning. University of Washington researchers first had to identify the gaseous components, an outstanding detective job in itself. Processes and equipment necessary to accomplish the treatment were then designed.

During cooking of wood to form cellulose pulp (as with any cooking) gaseous by-products are released. Presently kraft mills use all known and economically practical processes and equipment to reduce odors, but small, noticeable quantities of gases remain. These have not been removable with present methods. The important steps in the University's process which may result in almost complete removal of the odorous components are: (1) processing black liquor in stripping columns to remove odorous compounds, and (2) concentrating them in rectifying columns to such an extent that they can be consumed in a furnace.

Scientists emphasize that stacks and vapors still will be part of the industrial skyline even if the process is successful. The pilot plant is a "modest—but adequate" installation. Results still must be proved in full-scale operation.

The results gleaned from many of these research programs fall under the third major category of abatement—the indirect or corollary. Subheadings here could include: (1) by-products, and (2) new or altered processes.

Company research continually improves methods of using the components of spent liquor for commercial by-products thus reducing the waste load.

Typical of the progress in this field is the new $400,000 expansion program at Puget Sound Pulp and Timber Co., Bellingham. The company expanded their largest evaporator in order to obtain concentrated lignin. The new equipment creates a two-stage evaporator which approximately doubles its former capacity and a new spray drier to produce powdered by-products. Puget Sound Pulp also operates an industrial alcohol plant—the only operating plant of its kind in the United States—which converts wood sugars contained in sulphite liquor to 190-proof ethyl alcohol.

Spent liquor effluent from the alcohol plant enters the evaporators where it is concentrated for the manufacture of by-products. The concentrate, Lignosite, is used both as a liquid and a dry powder. In turn, it is used in the manufacture of a number of products including Q-Broxin, Viskal, Tacin, and Controlcal. These products go into the manufacture of linoleum cement, adhesives, leather tanning agents, and drilling mud additives.

Puget Sound Pulp supplies Lignostte to Monsanto Chemical Co. at Seattle from which is manufactured 50% of the nation's vanillin (vanilla). Lignosite is also used to manufacture chemicals for the agricultural industry which correct mineral deficiencies in plants.

Other companies such as Crown Zellerbach, Weyerhaeuser Timber Co., and Rayonier have similar commercial by-products.

Many sulphite mills are disposing of their spent liquor as a road binding agent. Lignin which holds wood fibers together works well in holding unpaved road surfaces together too. Last year spent sulphite liquor was applied to nearly 5000 miles of roads in Oregon and Washington. Companies which either sell or give away their spent liquor for road binding include Coos Bay Pulp and Paper Corp., Crown Zellerbach, Inland Empire, Publisher's Paper Co., Puget Sound Pulp and Timber Co., Scott Paper Co., and Spaulding Pulp and Paper Co.

Sprayed on road surfaces, the liquor forms a long-wearing crust which holds down dust. Concentrated liquor has been exported to other states for experimental use. It has a growing acceptance by city and country road departments, private contractors, and industry members find it useful on

their own logging roads. A professor at the California Institute of Technology has suggested use of spent sulphite liquor as a soil stabilizer for earth-fill dams if it could be waterproofed, that is, made less soluble.

Passing on from by-products to new or altered processes under the general heading of indirect methods of pollution abatement, let us go quickly through a number of illustrations of new and altered equipment. These include savealls, air scrubbers, settling basins, and so forth.

The Krofta Flotator recovers fiber that was overflowing to the sewer. The installation is located at the Hoquiam mill of Rayonier, Inc.

A Pacific Industrial Engineering Co.'s flotation saveall used by Pacific Coast Paper Mills serves the same purpose.

Weyerhaeuser's kraft mill at Everett has experimented with various recovery furnace flue gas pilot-plant scrubbers to determine if fly ash remaining after an electrical precipitator could be removed successfully. Results indicated that a spray-nozzle, venturi-throat scrubber would remove about 90% of the sodium salts from the flue gas.

A full-size, stainless steal scrubber has been installed and tested. Results indicated that plant scale unit normally removes about 90% of the fly ash.

There is a new settling basin of Western Kraft at Albany, Ore., which recovers 50% of the lime solids previously discharged. The basin settles out lime mud from all water used in the lime kiln showers and Peabody scrubbers.

A York "Demister" has been installed on a dissolving tank vent stack at Longview Fiber Co. It is used for the removal of dissolved chemicals normally vented to the atmosphere.

These are a portion of the many pieces of equipment in Northwest mills which are indirectly helping to abate air and water pollution, but they are indicative of the progress taking place throughout this region.

We have only touched lightly upon perhaps the most gratifying of all the industry's efforts—that is the fine cooperation extended to state agencies in an effort to protect and expand aquatic life.

The Oregon Fish Commission and Weyerhaeuser Timber Co. are working together to build an experimental natural "fish farm" on the east fork of the Millicoma River in Coos County to supplement and establish fish runs in coastal streams. Commission Director Albert M. Day said a 10-acre pond holding 500,000 salmon fingerlings will lower the cost of hatchery-raised fish by placing fingerlings in impounded water with a natural food supply. Weyerhaeuser installed culverts and other modifications at a cost of $5000 and is giving up an acre of tree-growing land. "In addition, the company has voluntarily spent approximately $8000 to modify the new channel bed as a fisheries protection measure," Day said.

West Tacoma Newsprint Com., and three state agencies—the Pollution Control Commission, Fisheries and Game Departments—have joined in cooperative water quality studies of Chambers Creek, a mile-long estuary used for up and downstream migration of steelhead reared in the Game Department hatchery. The mill and Western State Hospital (population 3000) discharge waste into the creek. West Tacoma retained the Academy of Natural Sciences of Philadelphia and Dr. Edward Wood, fishery pathologist. All parties participated in field and laboratory work. Downstream migration of yearlings was studied, as well as fish in live boxes and aquaria, to determine influences of wastes from the hospital and mill. Much of the Academy work was done at the State Fish Hatchery. Laboratory work is being completed, and reports compiled from this mutual study should result in a better understanding of the problem and its solution. Although the immediate results have been very significant, the mill has scheduled slimicide operations so that discharges take place only on outgoing tides when adequate dilution is afforded. As further protection, the hatchery has scheduled release of downstream migrants also at times when tides and dilution conditions are adequate.

Summing up, the Pacific Northwest mills share a vital responsibility in protecting our two great natural resources—wood and water. We have made tremendous strides within our industry in wood conservation and utilization.

The mills have accepted and met the challenge of protecting our water resources. We accomplish this through such direct methods as water monitoring, control equipment, research, and education. A variety of new and altered equipment indirectly protects the air and water.

Finally, we use what I have chosen to call incidental methods of reducing pollution in which we re-educate our employees to be conscious of water control and in which our decisions to install new equipment is based heavily on their potential as abators of pollution.

Clearly then, our future progress must be based not only upon new technological methods, but also upon our continued dedication to the philosophy of conservation. For it is only through the continued dedication to our role as conservers of water that we can make further breakthroughs into a problem upon whose ultimate failure or success rests our own future.

Presented at joint spring meeting of the Pacific Coast Div.—The American Pulp and Paper Mill Superintendents Association and the Pacific Section, Technical Association of the Pulp and Paper Industry held in Gearhart, Ore., May 22, 1959.

Empire State (Metropolitan District)

The following talk was given at the Ladies' Night meeting of the Metropolitan District which was held in New York, N. Y., May 12, 1959.

The Paper World of the Future

W. C. Kohlman

THERE's a movie still showing that some of you may have seen, "Around the World in Eighty Days." When Jules Verne wrote the story 87 years ago, traveling around the world in only 80 days was such a breathtaking concept that the installments of the fictional story made headlined news. Twelve steamship companies even competed to have the fictional traveler, Mr. Phileas Fogg of London, cross the Atlantic on their ships. . .a dilemma Jules Verne solved neatly by having Fogg buy his own ship.

But leave it to a woman! Within only 8 years Nellie Bly—a real-life newspaperwoman—made the trip and made it in only 72 days, 8 fewer than Verne's hero, and enough to convince the world that what had been just "that writer's fantasy" was really possible and true.

Another story of Verne's—"A Trip to the Moon"—was even more fantastic. But if you've been following stories about the training and preparation being given our candidates for the first trip into outer space, you'll agree that even "that" fantasy looks as if it will become fact before too much time rolls by.

Or maybe you saw these headlines some months ago, "LeMay Beats Clock and Calendar." Flying a jet from Tokyo to Washington, our Air Force Vice Chief of Staff set a speed record—arriving over our capital 32 min. *before* he left Tokyo. Of course he had crossed the date line. That figures to "Around the World in 44 Hours."

Thirteen Americans are now being readied for man's first flight into outer space! Sputnik and Vanguard in orbit circle our shrinking globe in little over an hour! So the first Phileas Fogg of manned satellites will go "Around the World in 80 Minutes!"

On the subject of headlines and our world of tomorrow: Bell Laboratory scientists had this to say about the telephone in a technical paper they recently prepared: "Ultimately,

C. W. KOHLMAN, American Cyanamid Co., New York, N. Y.

at the time of birth, each individual will be assigned a telephone number which he will bear through life just like his name. The telephone, similar to a watch, will be attached to his wrist. The front will be a transmitter-speaker, and the rear a three-dimensional picture of the individual to whom he will be talking. At any time when he wishes to talk with anyone in the world, he'll simply pull out the device —punch on the keys the number he wants. Then, turning the device over, he will hear the voice of the individual he is calling and see the face on the screen—in color and in three dimensions." Impossible? Sure it sounds startling but hardly impossible. For we are closer to such wonders than the man of 1872 was to a space ship. So who will say these goals will not be reached— and perhaps sooner than we think? The simple fact is this: Man can do anything that the mind of man can conceive!

What is the mind of man conceiving for paper in the world of the future? A few years ago I made a rash prediction. . . that in 1980, the per capita use of paper in this country might well reach 1000 lb. per person. It was, perhaps, an eminently safe prediction. . .for one thing, I'm sure that when 1980 rolls around, nobody's going to remember it, or care. But the fact is that in the past few years since I made the prediction, paper—and paper board—have been heading straight toward that 1000 lb. per capita figure. As I see it, our world of tomorrow might very well be a world in which paper is king!

What about the exciting ideas that have paced the growth of the paper industry so far? What about the exciting new ideas that will shape the paper world of the future? Yes, it could be a paper world. . . .Someone said (I think I saw it in an advertisement in a paper trade publication) that if this wasn't the space age, it could be more correctly called the paper age. There's something to that!

What's been done so far? What's made the paper industry grow so fast, and how has it grown? There are four great major concepts that account for much of the growth that paper has experienced.

First: the concept that paper could be a cheaper, more convenient substitute for the coarser or heavier types of textiles: cement and fertilizer bags, potato bags, flour and feed sacks, ice delivery bags, waste collection bags, mail sacks, auto-seat covers and rug backings. Second: the concept that paper could be used as a substitute for wood and other packaging materials, which has led to the replacement of wooden shipping crates and boxes, and has reduced the use of glass in the consumer distribution of milk. Third: the concept that our health can be improved, and cleanliness, comfort, and safety increased by the use of disposable paper items, such as tissues, paper napkins, paper towels, barber and beauty-shop towels and neckbands, hospital pads and bedsheets. Fourth: the concept of paper as a useful adjunct in manufacturing, industrial and construction fields: as industrial filters, as a base material in hard plastic sheets, such as Formica, in electrical equipment and machinery, for concrete forms, for tarpaulin covering of green concrete to insure proper curing, and innumerable other specialized uses. These are the concepts that have made paper one of the top, fastest growing industries in the United States.

That's today—But what about the years ahead? Anything can happen tomorrow—and tomorrow can begin to happen next year. Remembering that it took a lady—Nellie Bly—to translate a man's fictional trip around the world into fact. You ladies perhaps can go much further into the future and create a more complete world of tomorrow. In a world of such rapid change, we can't stand still—we must have new concepts. For instance, we have spoken of the tons of paper used as substitutes for coarse textiles, particularly in bags and sacks. But what about paper as a substitute for other textiles. What about paper clothing? Think of the fantastic potential of paper underwear alone. Can you imagine no more laundering, yet a sanitary cleanliness we can hardly even think of today? Can you imagine underwear that you just throw away when it's soiled? Consider the tons of paper that would be consumed by a population of 200 million in 1980 if this concept comes true. Of course, such paper would have to be soft, absorbent, strong when damp, elastic perhaps. . . .For women's underwear it might have to be thin—sheer like tea bag stock. It may be difficult to achieve all those characteristics—but not as difficult as building a space ship. And if the problem of disposable paper underwear is solved, why not disposable dresses like these experimental styles you're looking at? Why not a paper wedding dress or office outfit or bathing suits. Or how about disposable paper raincoats—the "wear-it-a-couple-of-times and throw-it-away-variety," which, incidentally, are available right now! All within the realm of possibility.

Or how about paper clothing for men? Today, paper slacks are being worn in some of our atomic research centers. They've been proved practical, just like the paper rain cape and paper baby carriage.

A paper world like that would use a thousand times more paper than we use today writing letters—an age-old habit which could even be obsolete in 1980, when we're all wearing our world communicating telephones on our wrists and when every business keeps its records by means of an electronic memory machine.

The things that I have just mentioned are not just wild concepts. Some have already moved from the concept stage to pilot experimentation. Of course, we'll have to learn much more than we know now about fireproofing paper before we can use paper for all these items of wearing apparel. But fireproofing paper is a less complex problem than launching an earth satellite. And then there's this about our world of tomorrow: it's going to be a world where people have leisure far beyond what we know today. If we continue only a modest 2% per year increase in productivity, our vacations are going to be longer and more people are going to enjoy them, while our expenditures for recreation amount steadily. So let's have paper sails for the tens of thousands of sailing craft that young and old Americans will be using in the new world of leisure. For that matter, why not a paper or paperboard boat? A boat to throw away each season—cheap—light in weight—no carting home in traffic. (Those family-size helicopters aren't being planned with too much trunk room. . .) And while we're at it, how about this extra leisure time and outdoor life, camping and the like. Paper shelter for the car or station wagon. . .paper sleeping bags. . .paper raincoat. . .These items are available too right now. As a matter of fact, pictures appeared in a recent issue of the Ford Co.'s *Station Wagon Living* magazine. And for that vacation cabin—why not one made of paper? Or paperboard. Durable for four or five years—longer if painted. Costing little more than a tent, but with the comfort of a real cabin. A dream? Yet that's exactly what we're looking for, isn't it? Dreams to make future realities.

Of course there's nothing very imaginative about a paper house. The U. S. Marines and the Army are experimenting in substituting paper housing for the two-phase tent and (then) heavy, uneconomic wooden construction that puts such a burden on our logistics today. The military conceive of paper houses that can be erected almost as cheaply and quickly as tents, and which can continue in service as base housing. In tomorrow's world, military housing could be, therefore, a one-step operation instead of the cumbersome problem it is today.

Fourteenth Paper-Plastics Conference

Edgewater Beach Hotel, Chicago, Ill.
Sept. 21–23, 1959

When paperboard was first substituted for wooden boxes we didn't try to make paper lumber and then saw it up and nail it together as wooden boxes were made. A new form was evolved. I am sure that every time a new use for paper is conceived and is made to come true, both the paper used and finished product will evolve their own form.

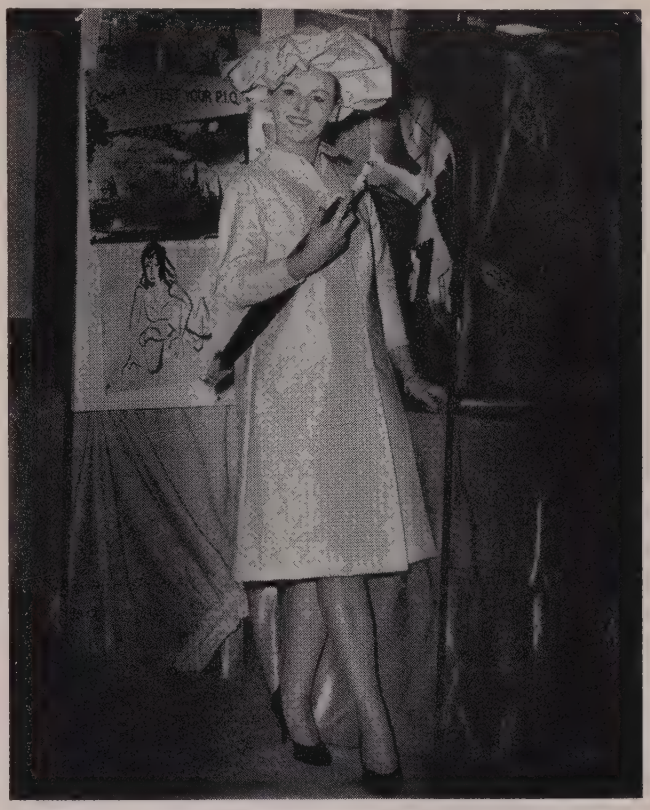
What about the potential in paper housing? Military use alone would run into enormous tonnages. Add to this the potential for recreational uses, for farm tool houses, for storage sheds, and you begin to get into very large figures indeed. And think of the possibilities that lie in paper housing in nations such as Mexico. Paper housing could make it possible to take a tremendous step forward in human welfare (at an approachable cost) compared to the expenditure for any other existing type of housing. Of course, before paper houses can go on sale at Sears Roebuck, we'll have to have at least one new characteristic in paperboard—durable stiffness, in spite of moisture. Once durable stiffness in paperboard is achieved paper housing won't be the only application to follow.

In the short time that we have tonight, I can't even list for you the new uses of paper that are actually in experimental form today, let alone even *begin* to explore our future world and the products it might well make use of. All I can consider is a way of life in the future that seems likely to the eyes of mid-century man. Think for a moment of only one part of tomorrow's way of life: sanitation. Conceive of cleanliness—but in terms that make our present standards seem as old-fashioned as the endless roller towel of 75 years ago. (Or was it only during this century that the roller towel gave way?) Actually, we could live in a disposable world in which every single household task involving dirt is performed with paper. Wiping towels, floor mops, polishing cloths, dust cloths, waxing cloths. . .every single one of them could be made of paper, and they would permit tomorrow's man to achieve sanitary standards of which we can only dream.

Imagine the selling appeal of chinalike appearing plates and cups and dishes that can be thrown away after eating. . . as superior in sanitation to today's dinnerware as the paper towel is to the textile roller-model, and just as possible as a widely accepted substitute. And while we're talking of paper plates, think of what they would mean in a wider sense. . .no more dishtowels, of course, and no dishwashing machines, either! If the automatic electric dishwasher is easier than hand scouring, one-use plates to be discarded right after a meal are obviously even easier and more in keeping with the trend toward simplified living. For that matter why not paper cooking dishes or paper pot-liners? Cooking surfaces would be completely clean—cleaner than they are today, even though housewives laboriously scour kitchen utensils. Preprepared frozen foods and meals cooked by cold-to-the-touch electronic stoves in moments. . .are these the meals of 1980? If they are, paper doesn't even have to have any new characteristics to be eminently practical for cooking in. It is possible today—or the first day that the first woman outdoes the Joneses and orders an electronic stove.

Briefly, the possibilities for paper in an age of experimentation are unlimited. Why not a paper body for tomorrow's auto—an all-over "top" to be changed during the life of the motor, to follow the seasons, the styles, and the particular needs of the owner. Such an auto—"fully convertible"—would probably be a new shape, maybe one that today's dreamers have in their imaginations already. Certainly, in no case will it be the car of today—or even a close relative. Even if it isn't directly within our province, we might all realize that tomorrow's personal transportation machines will, in no case, be like today's. Our diminishing oil reserves will force that issue in time.

In agriculture—why not tobacco shade paper—instead of tobacco shade cloth covering the miles of fields? We're using some paper right now on the farm for snow fences and emergency corncribs, and these planting caps are old hat. They've been around for more than 20 years. Our new sanitation for the home also has applications on the farm—paper wiping cloths for cows are already in being, but why not paper for the pails and containers into which milk production



Though showers may come her way, the smart lass can beat the downpour with the latest in paper—a disposable raincoat. Available from Disposables, Inc., the coat and specially designed rain hat were modeled by Debbie Johnson during the paper industry convention in New York.

goes? And paper liners for the cooling vats—all of them disposable, of course, so that our ever-scarcer farm labor force is freed from these time-consuming chores.

And in building construction, increasing use is being made of paper forms. Perhaps you know of the work being done to utilize paperboard cylinders in poured concrete to provide a lighter slab, with a naturally strong series of internal, arched air spaces.

In many fields, paper, I believe, can offer a challenge to more expensive materials, just as I suggested it might do in the case of steel for auto bodies. Paper is already being used in sanitation-conscious personal service businesses like beauty parlors. Some of our finest luggage—and our lightest—is paper, fabric-covered. Paper coasters are becoming commonplace, like paper hospital sheets and auto-claving bags. And what about an item for the head that's dear to the heart of every woman! Her chapeau! Made of paper? Why not! That's just what we thought at American Cyanamid and had a popular Manhattan milliner come up with these hats of the future, created of woven paper, and patterned after the headgear of the spacemen! We'll admit these hats are not necessarily what you'll be seeing on the first passenger rocket to the moon, but they express, at least according to William J. the milliner—and I quote him—"the marvellous new era of space and speed that we are entering." The hats, created of a "fabric" as new as tomorrow, symbolize the jet age. You may have seen this particular photo. . .one of the stage and screen star Celeste Holm modelling these space age paper hats in your local newspaper. The papers called the one on the right "Flying Disk" and the other "Turbo Turban." Or maybe you were looking in

on Dave Garraway's show the day Jack Lescoulie and actress Doretta Morrow showed what's ahead in the way of paper millinery! Their's was a comic stunt! But wait a minute, what's wrong with these hats all made of the same kind of

Actress Celeste Holm models two hats styled of paper at the Hotel Commodore, New York, simultaneous with opening of the annual convention of two segments of the paper industry. At left is a "Flying Disk" lace evening cap; at right is a mushroom-cloud-shaped topper called" Turbo Turban." Both were designed by William J., New York milliner.

woven paper, strawlike in appearance. Right now several hat manufacturers are exploring the possibility of using these paper "fabrics" in their 1959–60 lines!

And while we are substituting, why stop with paper millinery. How about those ladies' paper clothes featured on the front page of *Women's Wear Daily* a week and a half ago.

And, how about those glass milk bottles. A substitution, incidentally, that is by no means complete, with problems remaining in spotty consumer distaste for the wax used today. Why not *all* carbonated drinks—and beer—in paper containers? Why not paper "bottles" and paper "cans"? What material better than paper for any disposable container for any conceivable product?

I fear I've been too practical, however, too tied to today in my examples. You, perhaps, can go much further into the future and create a more complete world of tomorrow. When I started, I said that what the mind of man can conceive (I'd better change that right here and now)—what the mind of *wo*man can conceive—*Man* can do. I hope that the paper industry in 2050 and in 1980—and in 1960—will prove us out!

Presented at the meeting of the Metropolitan District of the Empire State Section of TAPPI held in New York, N. Y., May 12, 1959.

Empire State

The following paper was presented at the meeting of the Empire State Section of TAPPI held at Lake Placid, N. Y., June 13, 1959.

Vacuum Metallizing Techniques for Paper Combinations

K. C. Taylor

The inclusion of this subject in today's program on "Films and Foils" along with paper coating techniques is exceedingly timely. The day of economical volume production of vacuum deposited coatings on various papers is not yet here, but it is approaching. It will require 1 to 2 years more of intensive work to achieve its commercial breakthrough.

Vacuum metallizing is a very large field to cover thoroughly in such a brief period; however, I shall seek to present an over-all understanding of the process together with a status report of the technique as it now stands.

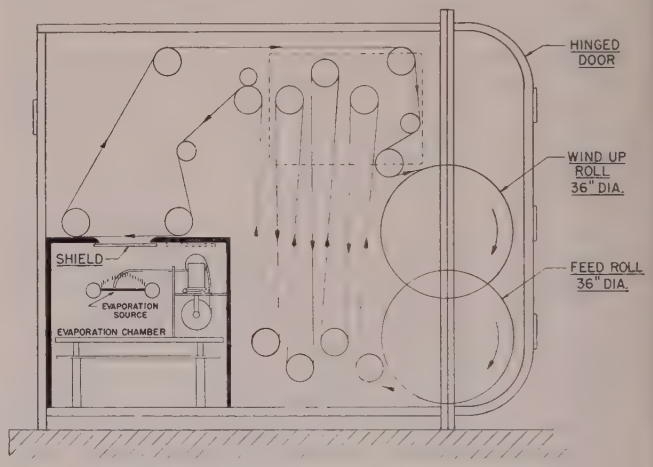
K. C. Taylor, Product Specialist, Vacuum Equipment Div., F. J. Stokes Corp., Philadelphia, Pa.

Vacuum metallizing is nothing but vacuum evaporation! The resultant product, the metallized coating, is distilled metal molecules embedded into paper fibers.

Like every other commercial process, this one has certain fields in which it excels. Metallizing's modern technology started about 1935 when it was first used, as it is today, for highly specialized optical coatings: i.e., for filtering, color correction, and reflection control on all types of lenses. The goal which concerns us here is to produce a paper product with the approximate reflectivity of laminated aluminum foil at 50% of the cost of aluminum foil, but maintaining the many advantages of an all-paper product.

It is first necessary to understand two basic fundamentals regarding this process:

Only extremely thin coatings are possible under normal, economic conditions. These coatings are normally measured in micro inches. On both plastic films and papers, a deposit of 2 to 3 micro-inches (i.e., millionths of an inch) of metal is normal. It is this precise thickness control which explains its use in optical coatings. In view of these extremely thin coatings, the product will therefore continue to perform as a paper because it is essentially, and continues to be, paper. (Incidentally, thicker coatings, of several thousandths of an inch, are being talked about for other fields and can be applied, but they are not yet to be considered truly economical.)



Outline of the vacuum metallizing process

To accomplish vacuum evaporation at significant or commercial rates, we must maintain both high vacuums and high temperatures of the evaporate. Neither of these conditions are particularly *inexpensive or easily obtained.*

Speaking broadly, vacuum evaporation is accomplished at an absolute pressure of less than 0.001 of a millimeter of mercury. This pressure is readily obtained today by vacuum technologists, but has only been available in these huge capacities for a few years. What this low pressure means is that on a volume basis every particle of water upon evaporation in a metallizer expands at a ratio of 1,000,000 to 1. Needless to say, expressed as cubic feet per minute of vacuum pumping load, the volumes incurred in the production of metallized paper are huge. I would like to think that this partly explains why vacuum coated plastic films have been available commercially for more than 5 years, while similar metallized papers have been notably absent. In brief, suitable vacuum pumping equipment, although in use in vacuum metallurgy for 5 years, is just entering this field.

COATING METHODS

There are three vacuum coating techniques of interest to us today:

Batch Coating

This is the evaporation of metals or metallic compounds from stranded tungsten filaments. The coating is measured

in grams of metal evaporated for periods of seconds. The total life of a tungsten filament in this process is 90 sec. This is a method used by custom coaters, who apply decorative coatings to such items as toys, automotive trim, etc. Its economic value is longer life at lower cost than electroplated coatings. It is of little value to the paper industry except for exploratory work.

Semicontinuous Coating

This is the present state of the art! In this process, a roll of paper of any size is placed within a vacuum chamber. The chamber is evacuated, the web of paper is drawn over a large bulk source of evaporating metal and coated as it passes. The vacuum must be broken when the web is exhausted and the machinery recycled. Normal running speeds are 500 f.p.m. Complete costs using this technique are predicted as $2.10 per ream. Please note this doesn't include the precoating. This method is satisfactory for plastic film and glassine manufactures, but is of less interest for heavier gage coating operations unless extremely large rolls of great length, giving long running times, are used.

Truly Continuous Air-to-Air Coating

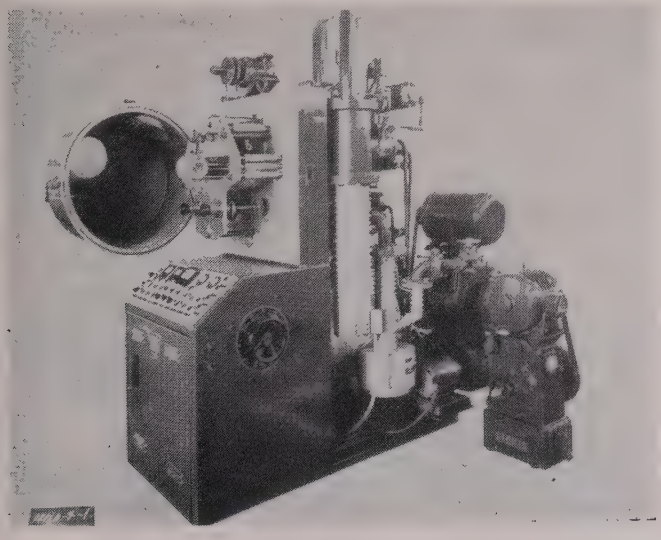
The third and final technique will be the use of truly continuous units with the web of paper passing from the air, through a seal, into the vacuum to be coated, and then back into the air again without interruption. Units of this type will be brought into operation this year.

Figure 1 shows a schematic view of a semicontinuous coater, typical of units which are being operated today. This unit can readily be converted to a fully continuous unit, i.e., an air-to-air machine, by adding a seal and relocating the feed rolls once the sealing mechanism is available. Figure 2 shows a 6-in. web capacity experimental coater. Note the relative size of the vacuum pumps in comparison to the coating chamber; this is somewhat typical. Figure 3 shows a 54-in. web capacity semicontinuous unit for plastic film coating only.

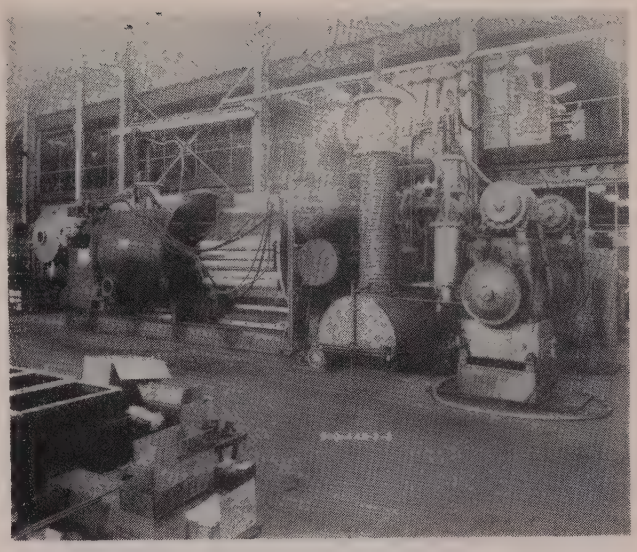
When we concern ourselves with the application and utilization of this process, we find a severe limitation in the form of available compounds that may be commercially applied.

Most of the noble metals are ideal except that they are out of reach on a cost basis. The more common metals are often unsatisfactory because of some unusual quirk or else they produce a dull appearance. Generally we end up with a choice between aluminum and zinc.

Zinc has been vacuum metallized for by far the longest time and is used exclusively today for the manufacture of capacitor tissue. Aluminum coatings, on the other hand, have an unlimited shelf life and far superior reflectivity. All



Metallizing unit



Installation of a vacuum coater

metal which is vacuum deposited on a substrate is just a succession of individual molecules and is not a truly homogeneous layer such as a wrought product. It is therefore inclined to be porous. Although it gives some barrier properties, they cannot be considered outstanding. The only true function of a metallized coating (other than its electrical properties) is its very high reflectivity which is approximately 86%. This has limited its use to either thermal properties (reflection) or decorative application.

PRODUCT APPLICATIONS

Continuing with our application review, we see that vacuum coating of plastic films is now practiced fairly extensively. I am told that vacuum coated vinyl film will appear in major quantities this Christmas as an improved form of Christmas tree tinsel. It will be fire-resistant, and more pleasant to handle than conventional foil tinsel. It can be easily pulled out of a bundle one strand at a time. Another functional use of vacuum coated plastic film has been for large superstratosphere balloon gas bags which are metallized in order to reflect the intensified sunlight and thus cool the bags. However, all of these plastic films fall into a fairly high price bracket.

The next category of application will be glassine vacuum coated with aluminum. This is now appearing as ice cream bags.

It certainly could be expected as the next logical extension of the process, because the density and surface texture of glassine makes it, in the eyes of a metallizing operator, a hybrid lying midway between plastic films and general coated papers. Difficulties are experienced with glassine due to the extensive, but small, dull spots which are caused by the inability of the supercalender to level the fibers at the bottom of the wire marks. This condition is improved, however, by either precoating or embossing the final product.

The third and final field of application (excluding fabrics) is coated papers of all types. Machines are being constructed to handle normal paper moisture contents of say $5^1/_2\%$, without a processing loss of greater than 0.2% (moisture). In order to obtain any function (such as reflectivity), the base paper stock, be it book paper, label paper, heavy weight kraft, or boxboard, will have to be precoated. This precoating will be required to provide smoothness to the base sheet and may provide other properties such as a moisture-barrier or a gas transmission-barrier. This precoating will be an absolute necessity. You must understand that vacuum deposition is the *highest quality method known for high-fidelity surface reproduction.* It is the means by which the master records of

phonograph recordings are duplicated into dies to permit unlimited copying in subsequent manufacture. Frankly, this high fidelity of surface reproduction seriously defeats our purpose, because machine finished papers will give only a mat appearance of low reflectivity.

MANUFACTURING PROBLEMS

Limiting the scope of our study of applications to the principal apparent field, that is, the deposition of aluminum on coated papers, we see two difficulties ahead, both quite severe. One of these falls into the equipment manufacturer's area; the other in the paper manufacturer's area.

The equipment manufacturer has a severe problem of metallurgy to overcome.

Molten aluminum must be superheated to 2700°F., which approximates steel melting temperatures, to get an effective or substantial rate of aluminum evaporation. At this temperature, molten aluminum is extremely active and will alloy and combine with all common types of crucible materials including graphite. It will either dissolve the crucible or form a contaminating by-product. Only this year has crucible life climbed from 2 to 12 hr. This certainly is not our final goal, but it is sufficient to commence industrial production.

The second problem, which is not yet solved, is a low-cost method of paper precoating. This precoating not only must provide mirror-smoothness to the surface, but also must not outgas excessively in terms of the total quantity of gas and the corrosive nature of this gas. Otherwise, the aluminum vapor about to land will be oxidized and dulled.

The easiest precoatings are completely stable lacquers or plastic films. They also add other properties to the base sheet such as barrier values, but they have several disadvantages: (1) relatively high cost and (2) plastic films alter ink reception, alter machine folding, and weight yield. If extensive printing is going to be accomplished on the given paper, then specialized coatings of a clay base will have to be used. Such clay coatings seem the best suited, for all-around usage but are yet to be developed.

In conclusion, we see the vacuum metallizing process which is commercially established in other fields, being adopted as a reflective coating on paper of all types by the intense development of specialized machines and precoating. Much work is underway; much more work remains to be done. However, it promises to be a reality, in 1960.

Presented at the meeting of the Empire State Section of TAPPI held at Lake Placid, N. Y., June 13, 1959.

RECENT BOOKS

Semichemical Pulp. Raw Materials—Chemistry and Process Engineering—Economical Importance. By R. O. H. Runkel, Professor and formerly Director of the Institute of Wood and Cellulose Chemistry at Reinbek, Germany, and K. F. Patt, Tornescher Hefe Gmbh, Guentter-Staib-Verlag, Biberich an der Riss. Germany, 1958. Cloth 6 × 8½, 209 pages. Price: DM 22.50 ($5.50).

Semichemical pulping methods are more than 30 years old. Their prospects today look brighter than ever before. Now two authors, the first one a famous pioneer in this field, present a complete and detailed discussion on semichemical pulp. After clear definitions and characteristics, we find history as well as dynamics of this important development in the USA and abroad. The main part deals with a description of processes, including bleaching methods. Properties, applications, and economics of semichemical pulps are added. The book finishes with prognosis for the USA and Europe. Preface and conclusions are translated into English. This publication will be a very valuable source for all persons interested in pulping and papermaking.

MANFRED W. LUTHGENS

Books Reviewed in this section may be secured from TAPPI, 155 East 44th St., New York 17, N. Y.

LETTERS TO THE EDITOR

With reference to Mr. Banfill's letter about TAPPI standard T 7m, he correctly brings out some of the limitations of the volume measurement of pulpwood. If the wood is knotty or crooked, it seems desirable to specify that for the measurement of the proportions of the wood, the void spaces and the bark, the photographic dot- or grid-count method should be applied to both faces of a rack of the wood, and the results averaged.

With a given expenditure of time and effort, it is an interesting question as to whether greater accuracy could be secured by evaluating additional photographs from both sides of a stack of rough wood, or by determining the average moisture content of the wood and the actual wet weight of the bark present, so as to permit a correction to be made to the gross weight of a shipment. It probably depends, as Mr. Banfill has indicated, on the kind and type of wood being measured.

JAMES D'A. CLARK, *Chairman*
Standards Committee

EMPLOYMENT SERVICE

POSITIONS WANTED

E468-59. Man with extensive experience in technical and production phases of wide variety of papers, research technical and production supervision work in hard sized as well as low density absorbent specialties. Desires position where there is latitude for application of personal efforts and experience in any of those phases of the industry.

E475-59. Chemical Engineer desires a responsible position in the application and use of fine and technical papers. Has developed papers for specific functional uses in coating and laminating.

E476-59. Technical Executive: 20 years experience in paper coating, adhesives, and specialty papers, seeks challenging opportunity.

POSITIONS OPEN

P844-59. Sales Application Engineer Wanted for Sales Department of converting machinery builder in New York State. Prefer mechanical engineering background. Varied, interesting, and growing product line in paper, film, and foil machinery. Offers excellent growth opportunity.

P854-59. Research Chemical Engineer for Research and Development in paper and new products. Advanced degree desirable. Experience in paper essential. Salary commensurate with education and experience. Southeast location. Please send complete résumé and salary requirements in first letter.

Paper Chemists

For challenging positions in the Paper Development Group of one of the leading suppliers of coatings and sizing agents to the paper and corrugating industries. Position involves product development of natural and synthetic coatings, sizing agents and adhesives and technical service work to customers. Have openings ranging from beginning position for inexperienced B.S. or M.S. chemist to responsible position for Ph.D. with broad or specialized experience in the manufacture of paper.

Growth and development opportunities with research minded company in expanding industry located in modern midwest city of 80,000.

For information send résumé to G. M. Prust.

A. E. Staley Manufacturing Co.
Decatur, Ill.

(P867-59)

MECHANICAL ENGINEERS

The Equipment Engineering Section of the West Virginia Pulp and Paper Company has immediate openings for Mechanical Engineers at their Research and Development Laboratories located at Covington, Virginia.

JUNIOR PROJECT ENGINEERS

Requires degree in mechanical engineering with or without experience. Age to 30.

SENIOR PROJECT ENGINEERS

Requires degree in mechanical engineering and experience in the machinery field or the paper industry. Age to 35. These positions require versatility, creativity, and a high degree of mechanical ability to do responsible project engineering work. Working field: Paper converting and finishing machinery. Willingness to travel between plants is essential. Salaries open.

Send complete résumé in confidence to:

Research Director

WEST VIRGINIA PULP AND PAPER COMPANY

COVINGTON, VIRGINIA

(P870-59)

ENGINEERS

CHEMICAL AND MECHANICAL

We have openings for young Chemical and Mechanical Engineers in our Technical and Engineering Departments. These men should have from 2 to 5 years' experience in the pulp and paper industry. These are good opportunities for professional growth with an expanding concern. Salary commensurate with experience.

Personnel Department
Riegel Paper Corporation
Carolina Division
Acme, North Carolina

(P875-59)

Chemical or Chemical Engineer, with 3–5 years' experience required by resin manufacturer to evaluate resins and assist in design of new products for applications in paper and paperboard. Knowledge of paper coatings and general resin applications to pulp and paper desirable.

Location Chicago. Reply to P872-59, Tappi, 155 East 44th St., New York 17, N. Y.

PAPER COATING RESEARCH

Advanced degree in Chemistry or Chemical Engineering with three to six years' experience in paper coatings, resins, and resin emulsions required. Ability to plan and organize project; writing of clear, readable reports is also important.

Successful candidate is expected to advance rapidly to Project Leader.

Send complete résumé in confidence to:

Research Director
West Virginia Pulp and Paper Co.
Mechanicville, N. Y.

(P877-59)

PROCESS ENGINEER

A recognized leader in the manufacture of enamel book papers is considering applications for two positions as Process Engineers.

Our Process Engineers are part of the mill technical staff working closely with production personnel on problems involving quality control, wastes and efficiencies. These engineers are assigned to specific areas in the mill such as Groundwood pulping, Stock blending, Papermaking or Coating, and are expected to provide guidance and technical advice to the operating superintendents.

Paper Technology, Chemistry or Chemical Engineering background is preferred.

Two or three years experience in Process Engineering or mill technical service work is necessary for this position.

These positions are located in Central Wisconsin.

Please give full details and résumé in your first letter so that interviews can be arranged. Send to P881-59, Tappi, 155 East 44th St., New York 17, N. Y.

Midwestern specialty pulp and paper mill desires a technical graduate to lead a research and development program. Two to four years experience essential with a knowledge of cotton linter pulps and papers helpful. Excellent opportunity for advancement. Send complete résumé of education and experience to P882-59, Tappi, 155 East 44th St., New York 17, N. Y.

P883-59. Technical Director—Integrated Southern kraft mill has interesting and challenging position open for a top technical man with organizational ability as Technical Director. This man will direct existing Quality Control, and be responsible for setting up a department having authority and control of product development, process evaluation and control, technical sales service, and development of operating and supervisory personnel. He should be capable of relating his technical know-how directly to practical pulp and paper making. Such a man would work with and report directly to the principal resident officer, and also work closely with the company's President in formulating major policy and direction of the company's objectives. All replies treated completely confidential. Submit complete résumé and snapshot.

P884-59. Graduate chemist, chemical engineer, or paper school graduate for development work in technical department of integrated midwestern paperboard operation. 2 to 3 years experience in paperboard or related industries required, preferably in coatings. Excellent work situation and advancement opportunities. Salary open. Submit detailed résumé to Personnel Director, The Ohio Boxboard Company, Rittman, Ohio.

CHEMISTS-PHYSICISTS-ENGINEERS

Young men with B.S. or advanced degree in chemistry, physics, chemical, mechanical, electrical engineering or pulp and paper technology. Excellent opportunity, interesting research and development work in papermaking, coating and finishing of fine printing papers. Location in Western Maryland well known for summer and winter recreational facilities. Previous experience desirable but not essential. Salary commensurate with background. Submit confidential résumé to:

Director of Research
West Va. Pulp & Paper Co.
Luke, Maryland

(P885-59)

WANTED

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NATURAL
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The Cellulose Water Flow Number

O. A. BATTISTA, A. C. PACEWIC, and C. J. BOONE

Utilizing equipment and experience relating to the hydrocellulose water flow number (*1*), a companion test has been developed for fibrillating cellulose fibers called the cellulose water flow number or CF number. The cellulose water flow number has been found to be a rapid measure of the fibrillation of special paper-grade rayons due to mechanical action, for example, by a Waring Blendor. It appears to reflect both the degree of fibrillation and the amount of fines produced; the procedure allows for the measurement of fines plus fibrillation, or the effect of fibrillation alone. It compares favorably in sensitivity with the Canadian Standard freeness as well as the measurement of surface area and specific volume as determined by the liquid permeability method (*3*). The cellulose water flow number is suggested as a practical control procedure for use in the pulping and papermaking industries, and in particular in connection with the use of special paper-grade fibrillatable rayons.

PROCEDURE

The cellulose water flow number is defined as the time in seconds required for 100 ml. of water to flow through a 4-g. sample on a calibrated CF thimble under a reduced pressure of 60 mm. of mercury after a standard fibrillating treatment in a Waring Blendor (usually 10 min.). With special samples, it may be desirable to characterize the fibrillation by measuring the CF number with increasing times of Waring Blendor treatment (e.g., 10, 20, and 30 min.).

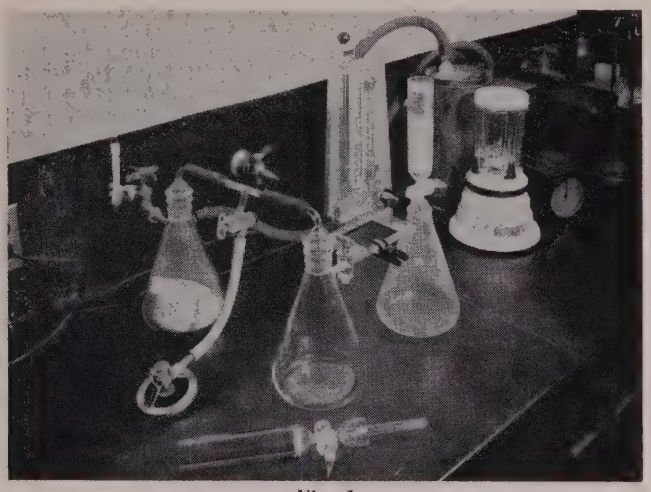

Fig. 1

The cellulose water flow apparatus is shown in Fig. 1. It consists of a special CF thimble (Ace Glass, Inc., Vineland, N. J.), two traps, a manometer, a needle valve to adjust the reduced pressure, a vacuum pump, and a Waring Blendor. The fritted glass disk and CF thimble have the same specifications as those used in making "Battista HF thimbles" (*1*) required for the measurement of hydrocellulose water flow numbers except that the height of the glass column has been increased to hold 100 ml. of water above the minimum draw-down line as a convenience.

The procedure used to obtain CF numbers is as follows. Four grams of oven dry sample are placed in a 500 ml. capacity Waring Blendor jar with 300 ml. of distilled water. The sample is treated in the Waring Blendor for a specified time. A 10-min. Waring Blendor treatment is used for normal CF numbers.

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The next step in the procedure is determined by whether the composite effect of both fines and fibrillation is desired, or whether the influence of the fibrils by themselves is preferred.

To Measure the Effect of Fines Plus Fibrillation

The sample is transferred directly from the Waring Blendor to the CF thimble. Application of reduced pressure is not made until the volume of water and sample in the thimble corresponds to at least 80 cc.; preferably, the thimble is filled with water to the top mark in the thimble. The time in seconds is then measured for 100 ml. of distilled water (between two calibrated circular markings on the thimble) to pass through the cellulose pad progressively formed at a reduced pressure of 60 mm. of mercury (±3 mm.). This flow time in seconds is the cellulose water flow number. When this procedure is followed, it is important to clean the CF thimble between each use to remove any fines which may collect in the pores of the filter. The cleanliness of the filter can be readily checked by measuring the ease with which water flows through it by gravity without a pad on it.

*To Measure the Effect of Fibrillation in the Absence of Fines**

In this case, the sample is transferred to a Buchner funnel equipped with an 80-mesh circular stainless steel or brass screen. This part of the equipment is not shown in Fig. 1. The pad that forms on the screen is washed well with distilled water to remove the fines, and it is then transferred directly to the CF thimble. The time in seconds for 100 ml. of distilled water to pass through the cellulose pad at a reduced pressure of 60 mm. of mercury (±3 mm.) is then measured. This flow time in seconds is the fines-free cellulose water flow number.

Since the degree of fibrillation and the amount of fines in pulp contribute to the strength of the paper which can be made from it (*3, 4*), it was felt necessary that the method developed should be a measure of both these parameters either compositely, or separately.

From the foregoing details of procedure, it follows that a clear statement as to whether the fines have or have not been removed via treatment of the cellulose pad on an 80-mesh screen should be made when listing CF number data.

Experimental

Prevention of "Packing" of Cake Prior to Applying Reduced Pressure. During preliminary work in developing a method for obtaining CF numbers, it was observed that the degree to which the sample was compressed in the CF thimble had an effect on the CF number. In determining CF numbers, the level of the water is allowed to reach but not allowed to go below the lower draw-down line on the CF thimble above the fritted glass disk. Since the height of this line controls the degree to which the sample is compressed, it was essential to determine the effect which the height of this line had on the CF number. The data obtained by varying the height of the draw-down line are shown in Table I. Rayon grade sulphite pulp was used to obtain these data. The sample was treated for various lengths of time in the Waring Blendor and the CF number of the treated sample was then determined using the various line heights.

The height of the draw-down line on the CF thimble is expressed in terms of milliliters of water at 25°C. required to fill the space between the fritted glass disk and the line. The data show that the higher the line on the CF thimble, the lower is the CF number. However, when the line corresponds to a volume of a least 80 ml. of water, the results are the same within experimental error. Thus, to obtain normal CF numbers it is necessary that a volume of at least 80 ml. of

* Arbitrarily, we have defined fines as particles of cellulose which will pass freely through an 80-mesh screen. Certainly, particles larger than this will not plug the pores of the standard CF thimbles in as much as their average size is of the order of 40 mu.

water be maintained above the filter before applying the reduced pressure.

Table I. CF Number as a Function of the Height of the Line Above the Fritted Glass Disk on the CF Thimble

Height of draw-down line, (ml. of H_2O)	*Waring Blendor time, min.* 2	4	6	8	10
48	73	143	224	278	402
66	62	122	168	239	311
82	55	105	155	221	291
100	54	107	153	206	261

Effect of Treatment Time in Waring Blendor. In order to determine the optimum time of Waring Blendor treatment to be used in obtaining a CF number, samples were treated for various times in the Waring Blendor and CF numbers were obtained on these samples. The results are shown in Table II.

The samples used in this study are identified as follows:

ICCA-1 . . . Cotton linters pulp, acetate grade
ICCA-3 . . . Prehydrolyzed sulphate tire cord pulp
ICCA-5 . . . Cellophane grade sulphite pulp
ICCA-7 . . . Paper pulp, regular grade
K-96-2 . . . Rayon grade sulphite pulp

Table II. CF Number as a Function of Time of Treatment in the Waring Blendor

Sample	*Time of Waring Blendor treatment, min.* 2	4	6	8	10
ICCA-1	18	26	38	58	85
ICCA-3	14	19	22	26	31
ICCA-5	135	356	718	1092	1680
ICCA-7	138	355	712	1278	2146
K-96-2	55	105	155	221	291

Microscopic observation of these samples indicated that the greater the degree of fibrillation, the greater was the CF number of the pulp. The degree of fibrillation increased with the length of Waring Blendor treatment. A 10-min. treatment in the Waring Blendor was chosen for normal CF numbers because this treatment gave data which gave significant differences between samples and at the same time good precision was maintained.

Comparison of CF Number with Specific Volume and Specific Surface. The liquid permeability method for the measurement of specific volume and specific surface of pulp fibers is one of the best methods available for the prediction of the strength of a paper sheet which could be made from a pulp. The data obtained by this method are functions of both the degree of fibrillation of the fibers and the amount of fines present. Table III shows data for both the specific volume and surface area obtained by the liquid permeability method and the CF numbers.

Table III. Comparison of CF Number with Specific Surface and Specific Volume by Liquid Permeability Method

Sample	*CF number, sec.*	*Specific volume, cc./g.*	*Specific surface, sq.cm./g.*
ICCA-3 Control)	14	2.55	8,400
ICCA-3 10WB	31	3.18	9,300
ICCA-5 (Control)	135	3.75	9,160
ICCA-5 10WB	1680	4.24	24,300

The specific volume and specific surface are especially sensitive to changes in the degree of fibrillation and the amount of fines in a pulp. The data shown in Table III indicate that the CF number correlates favorably with the specific volume obtained by the liquid permeability method. Also, the specific volumes and CF numbers are in the same relative order for the samples listed in this table. This indicates that the CF number appears to measure the same parameter as does the liquid permeability method so that added significance may be attached to this measurement in view of this correlation with a sound and independent method of measuring total surface area.

Influence of Fines on the Cellulose Water Flow Number. In order to determine the effect of fines on the CF number, samples of K-96-2 and ICCA-5 were made fines-free by classifying them in a Bauer-McNett classifier. Material passing through a 150-mesh screen was considered to be fines. CF numbers were then obtained on the 10-min. Waring Blendor treated classified samples. The results are shown in Table IV. These data show the fines influence the CF number,

Table IV. Effect of Fines on CF Number

Sample	*CF number, sec.*
Sulphite pulp (viscose grade)—classified	115
Sulphite pulp (viscose grade)—control	311
ICCA-5 (classified)	487
ICCA-5—control	1680

especially if their particle size is very small. It was for two reasons that the use of an 80-mesh screen on a Buchner funnel was decided upon as a basis for removal of fines: (1) particles retained by an 80-mesh screen are too large to plug the 40 micron or smaller pores in the filters, and (2) an 80-mesh screen corresponds to the screen-size used in the Canadian Standard freeness equipment.

Correlations between the Cellulose Water Flow Number and the Canadian Standard Freeness

A comparison of CF numbers with Canadian Standard freeness data is shown in Table V. The CF numbers for the

Table V. Comparison of CF Number with Canadian Standard Freeness Data

Sample	*CF number, sec.*	*Canadian Standard freeness, ml.*
ICCA-1 (control)	18	736
ICCA-1-10WB	85	689
ICCA-3 (control)	14	737
ICCA-3-10WB	31	749
ICCA-5- (control)	135	693
ICCA-5-10WB	1680	449
ICCA-7-(control)	138	672
ICCA-7-10WB	2146	385
Sulphite pulp (viscose grade) (control)	62	730
Sulphite pulp (viscose grade)—10WB	311	644

control samples were obtained on 2-min. Waring Blendor-treated samples. The CF numbers for the 10WB samples were obtained on 10-min. Waring Blendor-treated samples. The Canadian Standard freeness of the control samples were obtained on the untreated samples while the freeness data for the 10WB samples were obtained on 10-min. Waring Blendor-treated samples.

Correlation Between Cellulose Water Flow Number and Canadian Standard Freeness for Two Wood Pulp Beater Runs

Two beater runs were made using a Valley beater with a 5500-g. weight on two different furnishes—(1) bleached hardwood, and (2) bleached slash pine. Samples were tested for both Canadian Standard freeness and cellulose water flow number after progressive 10-min. intervals of beating. In

all cases, prior to determining the cellulose water flow numbers, the furnish was treated on an 80-mesh screen in a Buchner funnel to remove fines prior to measuring the CF number. The results are shown in Fig. 2. Using a semilogarithmic plot, good proportionality was found between the cellulose water flow number and the corresponding Canadian Standard freeness with time of beating.

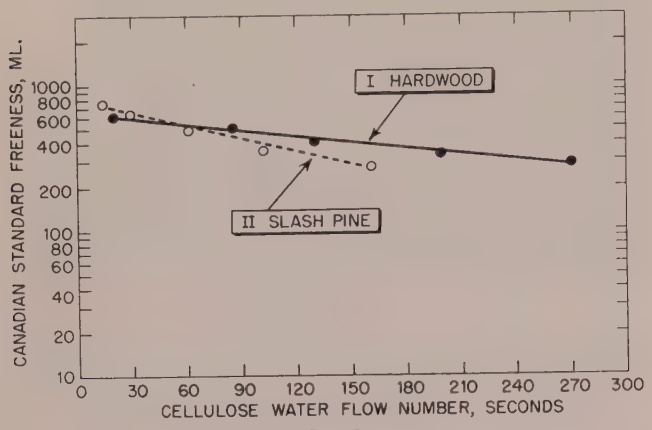


Fig. 2

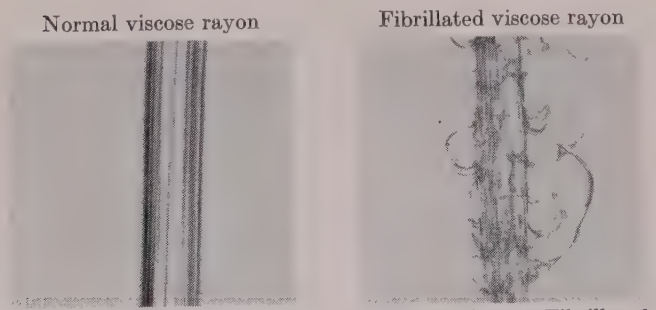
Cellulose Water Flow Number as a Measure of Degree of Fibrillation of Rayons

We were initially interested in establishing whether or not the cellulose water flow number would be useful as a yardstick to measure the extent of fibrillation of special paper-grade rayons as well as the strength of 100% rayon paper sheets made from fibrillated rayon fibers having increasing degrees of fibrillation. As the data in Table VI show, the cellulose water flow number and the tensile strength of 100% rayon paper sheets both increase with the degree of fibrillation. Microscopic observation of the rayon samples showed that these samples differed only in the degree of fibrillation (see Fig. 3). The data show that a direct relationship between the strength properties of a 100% rayon paper sheet and the CF number does exist. Thus, the CF number can be used to predict the strength properties of a 100% rayon paper sheet. The paper sheets in the above-mentioned instances were made from special fibrillatable rayon fibers which were fibrillated by means of a Waring Blendor action.

Table VI. Comparison of Rayon Paper Sheet Strengths with CF Numbers

Sample	*Breaking length, m.*	*Bursting strength, p.s.i.*	*CF number, secs.*[a]
A-155	958	9.0	56
B-555	1361	16.5	1240
C-1555	1963	23.3	2140
Control (unfibrillated)	0	0	8

[a] 80-mesh screening to remove fines was not used in the procurement of these particular cellulose water flow number data. Microscopic examination showed a definite increase in the amount of fibrillation with increase in tensile strength observed (see fig. 3).



Control—Unfibrillated cellulose water flow no. = 8 secs. 750 ×

Sample A-155—Fibrillated cellulose water flow no. = 56 secs. 750 ×

Fig. 3

SUMMARY AND CONCLUSIONS

1. It has been shown that the cellulose water flow number compares favorably in sensitivity with the Canadian Standard freeness and the liquid permeability method as a measure of fibrillation and/or increase in surface area due to a specific type of mechanical action.

2. The data presented indicate that the CF number is influenced by both the degree of fibrillation and the amount of fines in a sample. The procedure can be modified easily to measure the combined effect of fines and fibrils, or to measure substantially the effect of fibrils by themselves, and when the CF number is quoted, it should be stated whether it is fines-free or with fines; most useful data are based on figures obtained both ways.

3. Good correlation was found between the cellulose water flow number and the Canadian Standard freeness for two conventional wood pulp furnishes, as these furnishes were subjected to progressive beating with time in a Valley-type beater. Inasmuch as the procedure for the cellulose water flow number is quite simple, and the apparatus inexpensive, this method lends itself well for routine control purposes in the papermaking industry.

4. The cellulose water flow number can be used to measure the tendency of special paper-grade regenerated cellulose fibers to fibrillate and, in the case of some fibrillatable rayons at least, it can be used to predict the strength of sheets of paper made from rayons having varying degrees of fibrillation.

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Cold Caustic Pulping of Mixed Hardwoods

B. H. BAMER and H. MILLER

Stone Container Corp. realized as did several other producers of corrugating medium that the availability of straw as the principal ingredient was decreasing year after year. In spite of some information published by the United States Department of Agriculture, the use of the combine for harvesting wheat seemed likely to remain in our agricultural economy. Some members of the department felt that reaping still offered the wheat farmer the best utilization of his land and crops. In particular, the late S. I. Aranovsky was an enthusiastic proponent of the utilization of the extra production of straw which would result from this method of harvesting.

In general, combining became a more popular method and the availability and the price of straw delivered to certain mills became less attractive to the mills. Some mills, of

B. H. Bamer, Mill Manager, and H. Miller, Pulp Superintendent, Stone Container Corp., Coshocton, Ohio.

course, are still doing an excellent job of producing corrugating medium from straw. The problem is largely regional.

Stone Container Corp. was one company which felt that it was important to investigate other raw materials and processes for their production of corrugating medium. Hooker Electrochemical Co. made available to them the results of a cooperative study of cold soda pulping conducted at the Forest Products Laboratory at Madison, Wis.

The United States Department of Agriculture and other organizations had done spade work in this field. The laboratory findings had been favorable and the indications were that the quality of the pulp would be comparable to that of neutral sulphite semichemical pulps for the same end use. It was indicated at that time that the chemical costs would be lower but that the energy required for refining would be higher. There would be, of course, no cost for steam for digestion. It was felt that the possible savings in chemical costs and steam would have to be balanced against an increase in the mechanical energy for refining.

There has been much informative literature published about laboratory, pilot plant, and experimental operation; however, not much has been published about commercial production experience with this process.

Stone Container Corp. made their decision to try this process at their Coshocton, Ohio, mill for several reasons. The existing equipment could be used in the operation. The digesters and liquor preparation equipment such as liquid caustic unloading, storage, and dilution facilities were being used in the preparation of straw pulp. Secondly, the straw rotary digesters had pressure limitations which would require long digesting time for neutral sulphite cooking as compared to the usual higher pressure practices. This aspect was confirmed by a cooperative project conducted at the Forest Product Laboratory at Madison, Wis. Also, the availability of technical grade sodium sulphite was in question at that time.

Some additional equipment and operations were, of course, required to make the conversion. A woodroom was necessary. The design of the woodroom depended upon the type of wood to be used. Since the woodland area surrounding the mill produced considerable qualities of hardwood railroad ties, slabs were readily available. This fact led to the installation of a slab chipper. Conveying equipment to feed the chipper and deliver chips to the digesters was required. Since it was found possible to have the wood delivered to the mill by truck from nearby producers, the wood handling system was designed accordingly.

It was found that the area could also produce adequate quantities of mixed hardwood pulpwood. A ten-knife Carthage chipper was installed paralleling the slab wood system to the chip screen feeder bin.

Additions were also made in the primary refining equipment. A second single disk Sprout-Waldron refiner was added in parallel with the one used on straw and a S-W Anderson Duo-Press was installed ahead of the primary refiners.

An additional requirement was a means of cleaning the stock entering the headbox. A primary, secondary, and tertiary system of Bauer Centri·Cleaners was installed. No screens were used originally, but a Black-Clawson Selectifier screen has since been added to eliminate unrefined chips which, at times, reached the paper machine.

Experimental runs of N.S.S.C. pulp have been made recently with the existing equipment using pulpwood. A good grade of pulp was produced. The average C.M.T. lay in the normal range of the cold caustic sheet. There was no appreciable difference in the power required for refining. Perhaps, this results from the maceration the chips receive in the screw press ahead of the primary refiners.

WOOD

As stated, slabwood was readily available in the vicinity of the mill as a by-product of cutting railroad ties. The species are predominantly oak.

With wood utilization in mind, slabs were the principal wood used until this spring. Since then, mixed hardwoods have supplanted slabwood, as the result of a program intended to give a practical comparison between the two types of wood.

Slabwood is characterized by its high sapwood and bark contents and relatively low moisture content as compared with pulpwood on the as received basis. However, mill experience indicates that slabwood requires a relatively high caustic concentration and consumption as compared with pulpwood. There also appears to be a difference in chemical reaction of the respective types of wood with sodium hydroxide solutions. Observation of the soaking of slabwood chips in glassware shows extensive precipitation of some soluble components of these slab wood chips which has not been observed with pulpwood chips.

The desirable properties of slabwood are accompanied by the problem of separating and disposing of the larger percentage of bark. The removal of bark from pulpwood has been a problem of much concern to manufacturers of pulp for many important grades of paper and paperboard. Generally, the removal of bark from hardwood pulpwood has been a vexing and expensive operation. In some instances, modern methods of bark removal have proved to be satisfactory and economically sound, but for many mills bark removal has been found to be an expensive and troublesome operation. Some manufacturers have to be sure their products are almost entirely free of bark. With others, the questions of cost of removal, chemical consumption and other factors are considered.

Some components of the bark undoubtedly contribute some useable fiber to the finished product, other elements of the bark are a nuisance to the paper maker. At this point, Stone Container is not prepared to answer definitely the question of the economics of the bark accompaning the wood which involves the amount of chemicals consumed by it during digestion, its contribution to the usable furnish, and the cost of removal of its definitely undesirable components. The company's experience with the removal of these undesirable components of the bark by means of centrifugal action in Bauer Centri·Cleaners using primary, secondary, and tertiary cleaners has been satisfactory as far as cleanliness of sheet and operating qualities are concerned. Evaluation has not been made of the relative cost of chemical consumption and cleaner operation on one hand and the probable cost of debarking on the other, but it is felt that in the present state of the art of bark removal, the use of stock cleaners is satisfactory.

PROCESS AND EQUIPMENT

Incoming truck loads of wood are received and weighed. The wood is then banded with steel straps and driven to the unloading area. There, these unitized bundles are transferred from the truck to the storage piles by fork trucks.

These units are taken from storage to live bottom dry decks where the bands are removed and the slabs or logs are hooked onto a chain conveyor. The conveyor carries and elevates the wood to the chipper spout. From the slab chipper the chips are blown to a cyclone separator and discharged into the feeder bin of the chip screen.

The oversize rejects of the screen are returned to the slab chipper by belt conveyor, the fines are blown to a separator and bin. The accepted chips are taken by screw conveyor to an attrition mill fitted with dog-tooth plates serving to break cards and reduce slivers. The chips are then conveyed by screw and drag conveyors to the digesters which are fed by a traveling chute.

After a digester is charged with approximately 22,000 lb. of chips the caustic solution is pumped into the digester through the open head. The digester is then closed and rotated for

$2^1/_2$ hr. At the end of this time the remaining liquor is blown by compressed air from the digester to a used liquor storage tank having a capacity of 20,000 gal. The head of the digester is then removed and the chips are discharged by rotating the digester. The chips are discharged onto a drag chain as the former straw furnish was.

The chips are hooked into the original digested straw conveying system and discharged into a surge bin fitted with a live bottom screw discharge. The level of the bin is automatically controlled. This discharges into a variable speed screw conveyor which is manually controlled to maintain proper load on the screw press and primary refiners.

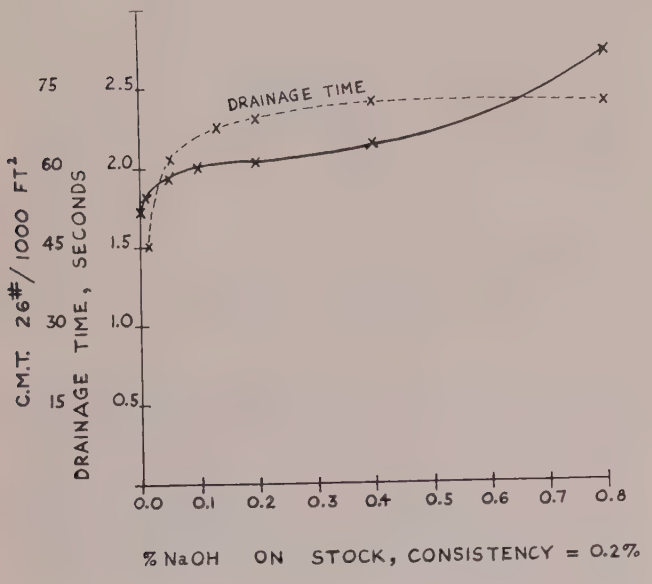

Fig. 1

In the primary refiners control is maintained by hourly tests of consistency and freeness. Wood stock from these refiners is discharged by gravity into a chest. From this chest the pulp is pumped to a consistency regulator.

The discharge of the regulator is then metered and combined with metered slush container stock and passed through two jordans into the machine chest.

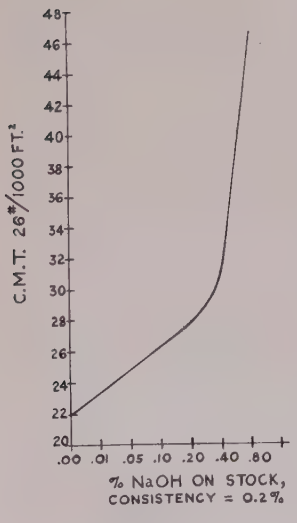

Fig. 2

The stock from the machine chest is pumped back to a consistency regulator in the refining room. From this regulator the refined stock is metered and flows by gravity into the suction side of the fan pump.

The discharge of the fan pump passes through three primary centrifugal cleaners and a selectifier screen to the headbox of the paper machine. The rejects of the primary cleaners are pumped through one secondary cleaner to the fan pump suction. The rejects from the secondary are diluted and pumped to two small tertiary cleaners. The accepted stock from these is returned for secondary cleaner dilution and the rejects flow by gravity to the settling basin where they join the expressed liquor from the Anderson screw press and ashed from the steam generator.

The effluent from this basin which receives these three wastes is practically free from settleable solids and is as neutral as the local ground waters.

CONCENTRATION OF STEEPING SOLUTION

Laboratory steeping of chips showed considerable variation in the rate of penetration, as judged by the time required for the chips to sink. The denser species of wood were penetrated more slowly. Increasing caustic concentration retarded penetration, while increasing temperature accelerated penetration.

These observations indicated that a solution concentration of 5 to 5.5% at 90 to 100°F. would produce a fairly rapid penetration of most of the species of wood used. Mill operation confirmed this, but showed that somewhat higher concentrations improved the ease of defibering the chips of some of the denser woods, such as oak.

It seems, then, that while the higher concentrations retard penetration, probably as a result of greater swelling of the exterior of the chip, they, again as a result of greater swelling, promote ease of separating the fibers. However, the retarding effect was found to reach a point where some chips had untreated centers at the end of a feasible period of steeping.

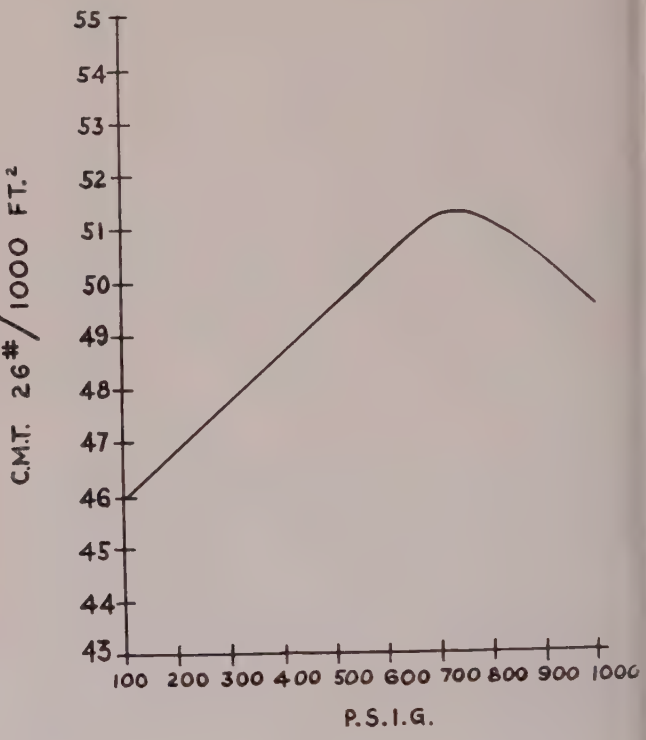

Fig. 3

A further effect of caustic concentration, which influences consumption, is that the higher the concentration the greater the amount of unabsorbed solution clinging to the drained chips. In the subject mill's system this is wasted.

These are probably the three most important considerations in determining a suitable concentration of the steeping solution. Where the unabsorbed solution is fortified and reused, high liquor-to-wood ratios can be used. Thus, the consumption requirement places no practical limitation on the selection of a concentration.

EFFECT OF VARIABLES ON PAPERMAKING AND QUALITY

Realizing that there are many factors which determine the quality and production of paper from the log to the reel,

efforts were made to recognize and control the most important variables. Attempts to find these relationships by direct tests in mill production produced some confusing data. Therefore, research projects were set up, hoping to find some fundamental conditions which would be guiding in mill practices.

Studies were made of the effect of caustic concentration and temperature upon the penetration of wood chips. Investigations were also made of the effect of caustic soda concentration in the headbox stock on C. M.T. and drainage time. The effects of wet and dry pressing upon C. M.T. were also studied.

We wish to acknowledge, at this point, information furnished to us by the V. D. Anderson Co., of Cleveland, Ohio, concerning their laboratory findings on the relations of caustic solution concentration, consumption, temperature of steeping solution and C.M.T.

The mill laboratory's work was mostly done with stock taken from the paper machine's headbox. These specimens were taken in sufficient quantity at one sampling to make a series of tests on identical fiber. Where the effect of caustic concentration was a variable being investigated, the stock

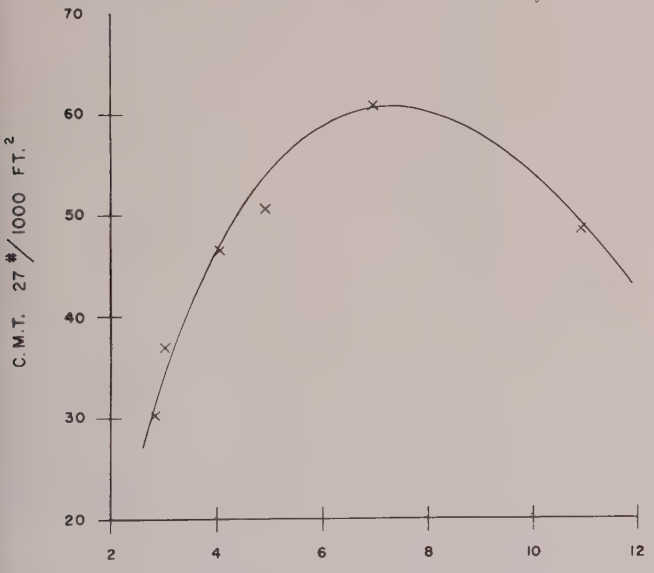

Fig. 4

was drained and washed three times on filter paper. It was judged by the rate of filtration and by titration that the residual alkalinity of the fiber was negligible for the purpose of the tests.

Portions of the washed stock, calculated to form a handsheet having a machine dry weight of 26 lb. per 1000 sq. ft., were diluted to a volume of 3 l. for the sheetmaking operation in a Williams 8 by 8-in. sheet mold. This dilution resulted in a consistency of a little less than 0.2%. Amounts of caustic were added to the separate stock preparations to give a series ranging from 0.0 to 0.8% NaOH concentration.

Specimens thus prepared were made into handsheets. The drainage time was recorded and the C.M.T. values of the finished sheets were determined. The results are shown graphically in Fig. 1.

Both the drainage time and the C.M.T. rise sharply from 0.0 to 0.1%. Beyond this point the rate of water removal is still considerably reduced with relatively little improvement in C.M.T. As with freeness, a compromise point must be found which will give satisfactory quality without unduly reducing production.

Figure 2 shows the effect of caustic concentration on the C.M.T. of handsheets prepared from waste container stock refined separately. The increase in C.M.T. shows that this effect is not peculiar to cold soda stock alone.

Figure 3 shows the relation of mechanical pressure on wet freshly formed handsheets and the resulting C.M.T. of the dried sheets. The values of pressure shown are the readings

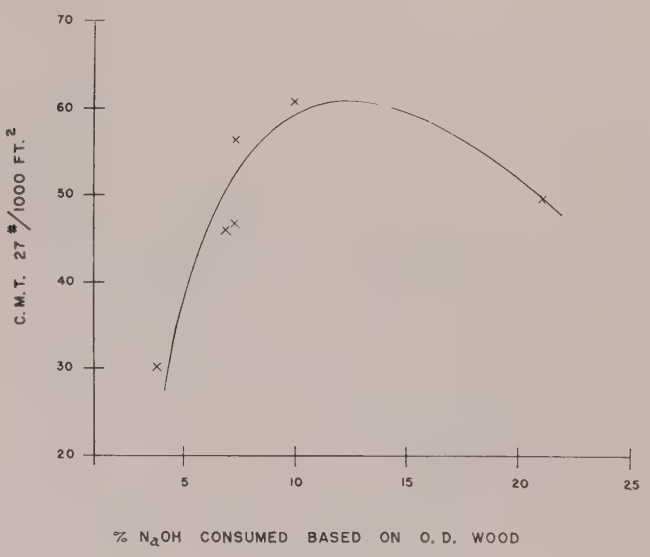

Fig. 5

of the hydraulic pressure gage of the Williams sheet press used. The pressure per square inch on the sheet is presumed to be about one eighth of these readings. The maximum strength exhibited at about 700 lb. may lie within the range of conventional paper machine presses, since some observations of paper machine operation have indicated that an increase in press loading may reduce the C.M.T. although no crushing is visible. While no data are presented, it was found that pressing of dried sheets reduced the C.M.T.

A type of data more directly related to mill operation is

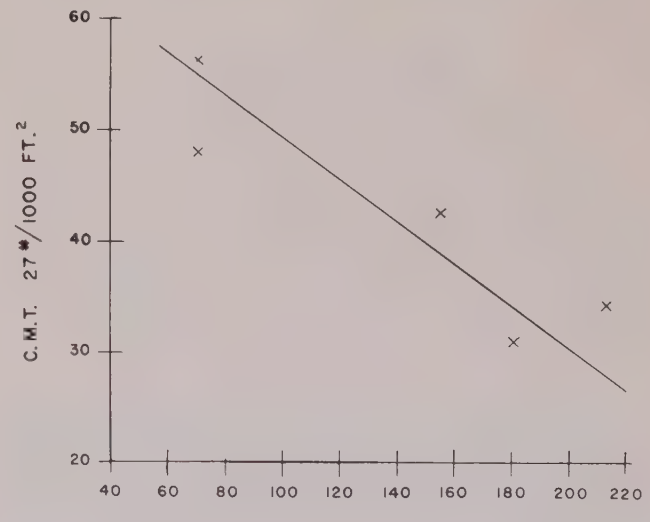

Fig. 6

shown in Fig. 4, 5, and 6. Figure 4 shows the effect of caustic concentration of steeping upon C.M.T. The maximum C.M.T. appears at a concentration of 7% NaOH. Figure 5 presents the relation between sodium hydroxide consumed and C.M.T. The maximum C.M.T. occurs at about 12%.

These data agree fairly well with mill experience considering the influence of wood species and the effect of operational differences between pilot plant and mill.

Figure 6 shows the effect of temperature of soaking on C.M.T., other conditions being constant. An approximately proportional drop in C.M.T. accompanies increase of temperature. Mill experience seems to confirm this effect also.

CONCLUSIONS

The equipment of Stone Container Corp. wood handling and cold caustic pulping systems have been described.

At this time it is felt that the conversion from the use of straw to the use of wood treated by the cold caustic process was a reasonable project. The conversion was made at a moderate expenditure and the results have been satisfactory.

Experimental data have been presented which seems to indicate that the use of sodium hydroxide in the cold soda pulping process serves two functions. First, it swells the wood and softens it for the mechanical separation of its component fibers. In the digestion phase of the operation the caustic concentration of the steeping liquor should be determined by its ability to penetrate and soften the wood so that steeped chips can be readily defibered. A second function of sodium hydroxide seems to be to condition the fibers for the papermaking operation to produce satisfactory quality of the finished sheet.

It seems that the principal function of caustic soda in these two respects is concerned with its ability to swell fibers and soften their exteriors. Pilot plant and mill experience can be interpreted that chemical reaction can be detrimental, as evidenced in Fig. 6 showing the effect of increasing steeping temperature. These conditions point up the importance of controlled washing between the operations of digestion and papermaking. It seems that a suitable degree of washing can be accomplished in a screwpress. The reduction of alkalinity of the expressed pulp depends in part upon the mechanical operation of the press and, also, upon the nature of the wash water used.

In planning for the future, consideration is being given to a trial of continuous cold-caustic treatment.

Presented at the Twelfth Alkaline Pulping Conference of the Technical Association of the Pulp and Paper Industry, Hot Springs, Ark., Sept. 23–25, 1958.

Evaporator Corrosion

HAROLD E. JACOBY and
HENRY G. LANKENAU

Corrosion problems experienced with pulp mill black liquor evaporators have been quite varied. The following information will describe typical cases illustrating the extremes and correlating these to the mill liquor system. We will also point up the problems that have arisen due to boilout cleaning methods employed.

In some mills, evaporator corrosion has been practically nil, while in others, corrosion has been very severe.

For example, mill A, having a six-body quintuple effect evaporator, went into operation in 1946. The evaporator was of all carbon steel construction, with the exception of type 304 stainless steel tubes, type 304 clad tube sheets, and clad vapor body section of no. 1A and 1B bodies. Number 5 effect was retubed with carbon steel tubes 6 years later; no. 3 effect 7 years later; no. 4 effect 8 years later. A new no. 5 body was installed in December, 1949, converting the unit to a seven-body quintuple effect. This new no. 5 body was retubed after $5^1/_2$ years in July, 1955. Number 2 effect was just retubed in July, 1958, giving 12 years life.

As for piping and general corrosion, the original plug cocks of ni-resist and about 98% of the original piping are still in use. Only two or three small sections of pipe were replaced at no. 1 effect. The only repairs on the vessels were the baffle tie downs on the product flash tank and the internal condensate flash baffle in the new no. 5 body.

This plant produces 95% bleached stock at a TAPPI permanganate number of 19. Prior to 1949, sulphidity was maintained at about 28%, since 1949 sulphidity is closely

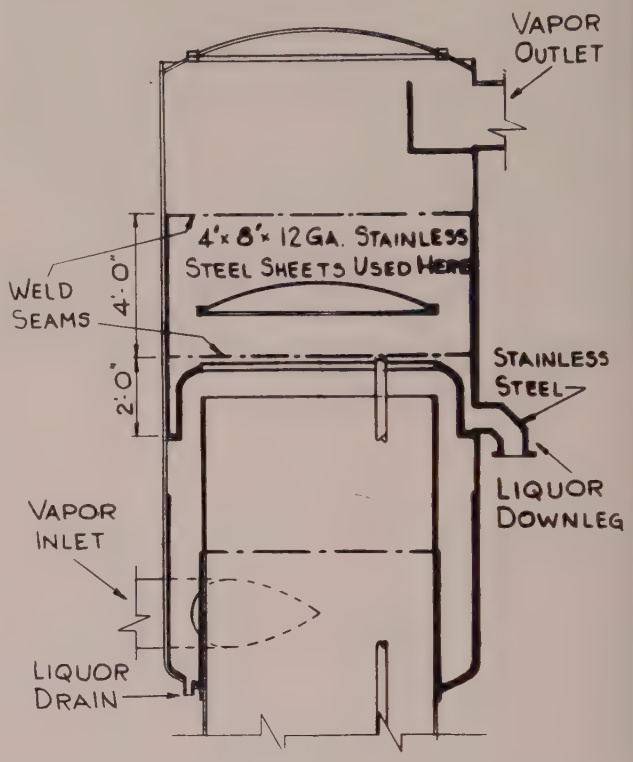

Fig. 1. Section through vapor body-heating element

controlled at 20 to 22%. Furthermore, very little elemental sulphur is used. Two types of pulp are made: one of 100% jack pine and the other of 50% jack pine and 50% poplar (cooked separately and blended). Total production is about evenly divided between the two types of pulp.

Only one acid wash was ever used on the evaporator, that was about 15% H_2SO_4 inhibited with salt cake. Water washout is given to liquor side only as a result of start-up and shutdown. Water washout on vapor side whenever opportunity permits. A 15% caustic wash (not white liquor) is given the vapor side of all effects except no. 1 every 2 months. Caustic is recovered through weak wash system of recausticizing plant.

One interesting feature of this mill is the surface condenser which was furnished originally with steel tubes, chromium plated on vapor side. Difficulty was encountered with water scale requiring frequent drilling. The condenser was retubed with Inconel in 1947. At that time Inconel was the lowest in cost. To date there is no sign of corrosion or deterioration of the Inconel.

Now for the opposite side of the corrosion picture. Mill B put a quintuple-effect evaporator into operation in June, 1955. Number 2 effect was retubed with type 316 stainless tubes in November, 1955, after the carbon steel tubes failed after approximately 3 or 4 months operation. Furthermore, the no. 2 vapor body, deflector and heating element projection all showed corrosion and erosion areas after the same short operating interval (see Fig. 1).

On all main walls of the vapor head, the metal failure left a very rough surface in the form of islands in the parent metal with the valleys between these islands about 3/32 in. deep. The deflector and heating element projection showed similar attack except that the islands were not so numerous. In

Harold E. Jacoby and Henry G. Lankenau, Chicago Bridge & Iron Co., New York, N. Y.

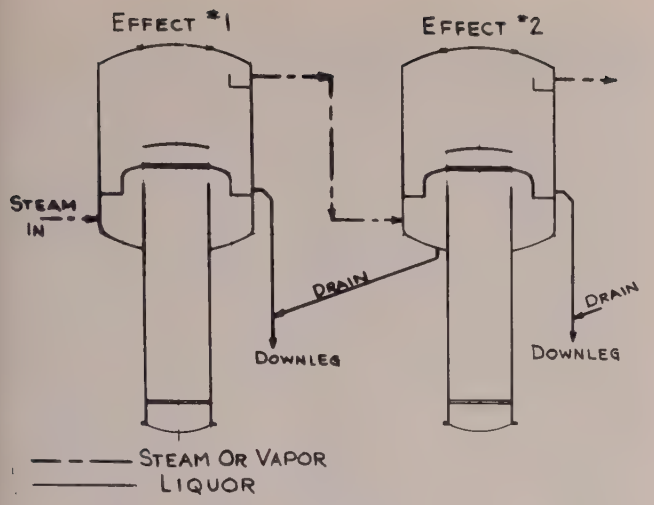

Fig. 2. Piping flowsheet

other words, metal removal was more uniform. A stainless steel insert-type lining was installed in the body to correct this trouble.

Since that time maintenance history on this evaporator has been as follows:

January, 1956 (6 months)—Liquor piping from no. 2 to no. 1 effect replaced with 316 stainless. Section of no. 1 vapor piping replaced with 316 stainless steel.

May, 1956 (11 months)—Number 3 effect retubed with 316 stainless after failure of carbon steel tubes.

August, 1956 (14 months)—Number 2 and 3 effect separator drain lines replaced with 316 stainless. Number 2 effect liquor piping by pass to product flash tank replaced with 316 stainless steel.

October, 1956 (17 months)—Liquor piping to and from product flash tank replaced with 316 stainless. Vapor header from no. 1 and 2 effects replaced with 316 steel.

May, 1956 (23 months)—Number 2 vapor body lined with 316 stainless at vapor outlet.

November, 1957 ($2^1/_2$ years)—A section of no. 1 separator replaced with 316 stainless and shell strengthened.

Finally, in May, 1958 (not quite three years) the entire no. 1 separator integral with no. 2 body was replaced with a 304 stainless clad shell and solid stainless inner baffle (Fig. 2).

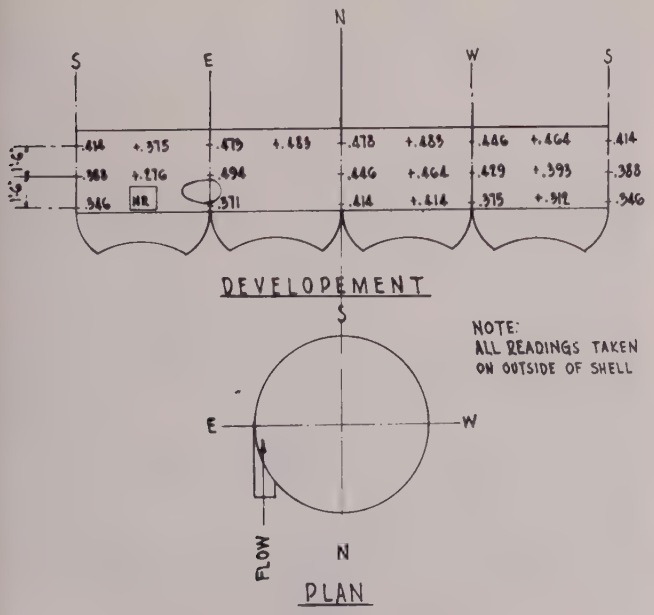

Fig. 3. Audigage readings

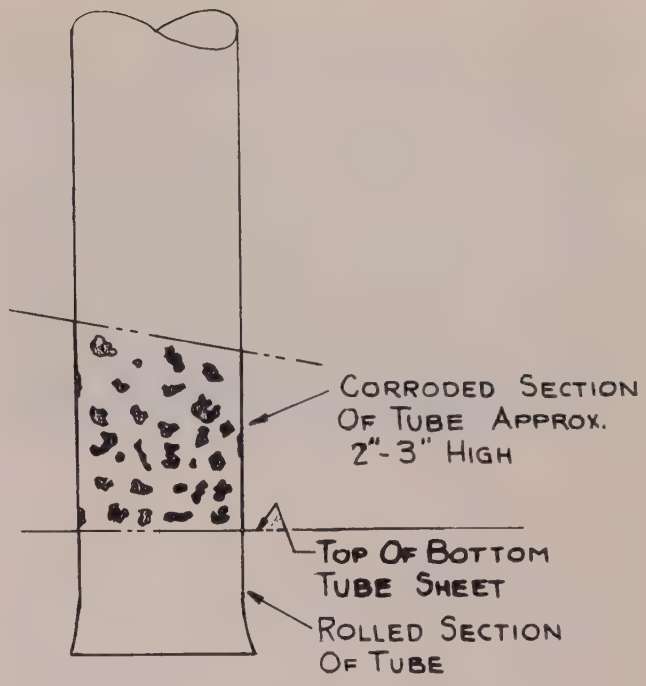

Fig. 4. Section of tube failure

This work was necessitated by corrosion as indicated by audigage readings (Fig. 3).

This plant is producing bleached grade pulp with average permanganate number of about 22. Production in 1957 averaged about 85% soft wood, 15% hardwood. Softwood varies from 100% to below 20% pine.

Sulphidity on white liquor in 1957 was 26.8% and sulphide on weak black liquor in the past year varied from 7.3 to 11.3 g. p. l. calculated as Na_2O.

It should be mentioned that this mill uses synthetic sulphide from their chemical plant as a means of maintaining sulphidity. Recent inspection shows that tubes in no. 4 effect are thin, also the no. 1 vapor body will probably require stainless lining in upper half of the shell.

New and severe corrosion problems are being presented by mills producing dissolving grade pulp. This has necessitated use of 304 stainless clad vapor bodies and bottom liquor boxes as well as stainless tubes, downtakes, and liquor piping for the first three effects. For example, carbon steel downtakes in no. 1 effect lasted approximately 6 weeks and cast iron control valve bodies gave about the same life. Number 1 and 2 vapor bodies show deep erosion areas in the carbon steel areas above normal clad sections.

Type 304 stainless steel tubes have proved to be very satis-

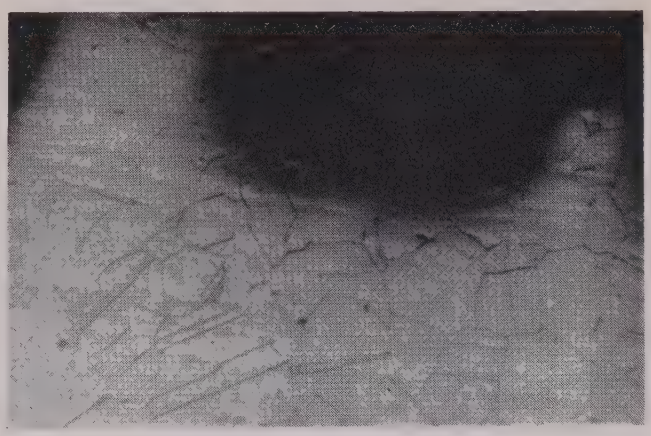

Fig. 5

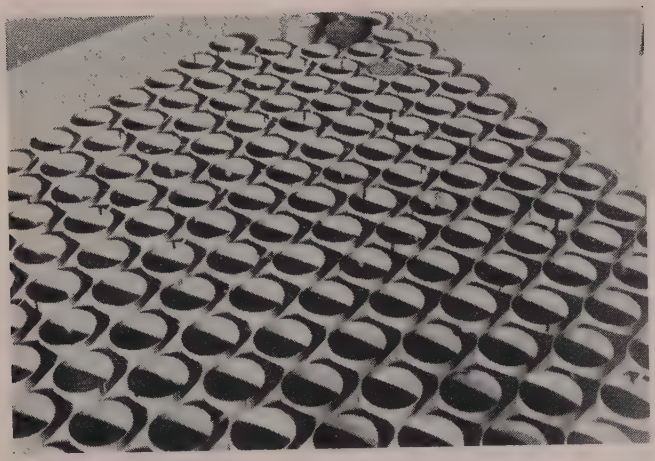

Fig. 6

factory. However, even here there have been occasional failures. For example, mill C installed 304 stainless tubes in no. 5 effect of a quintuple effect in 1949. In 1956 they were rather surprised to find eight or ten of these tubes leaking during a routine hydrostatic test. These tubes were removed and examined (Fig. 4).

At the time of removal, several inches of iron scale (presumably from the shell wall) were noticed at the point of tube failure. Chemical analysis of the tube material showed normal percentages for 304 stainless. Water extraction test failed to show signs of chloride or sulphate ions. Wall thickness of tube portion not attacked was 0.062 in., standard mill tolerances of 16 gage tubing are 0.065 ± 0.005 in. It is, therefore, evident that the tube had suffered very little loss in wall thickness from general corrosion.

The report states: "Since the corrosive attack was apparently confined to only one side and the lower end of the sample tube, there is reason to believe that the corrosion was

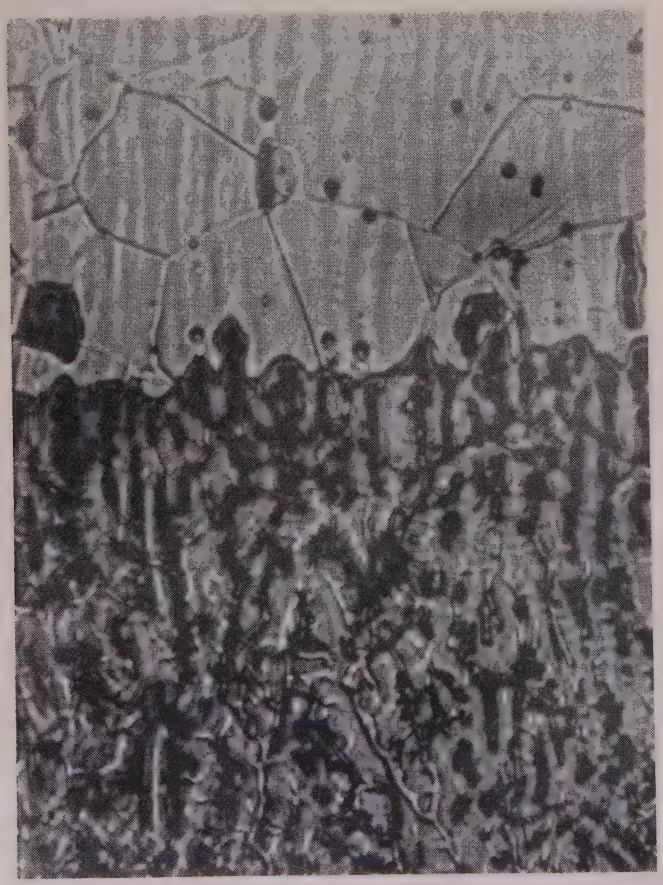

Fig. 7

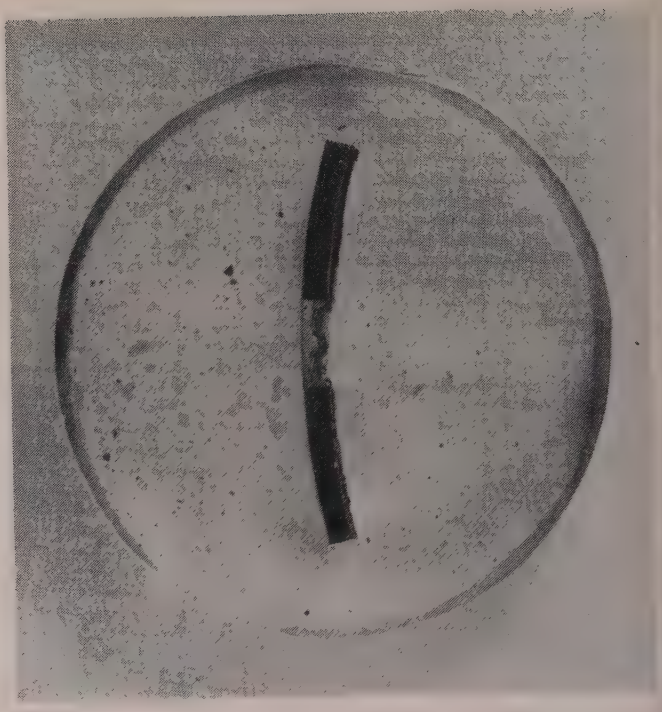

Fig. 8

caused by conditions which were local, i. e., peculiar to the lower portion of the effect, in nature. The accumulation of scale at the bottom of the effect where the corrosion occurred could, in time, build up and produce a concentration cell effect on the O. D. surface of the tube. This would result in corrosion similar in nature to that exhibited by the sample."

Figure 5 shows concentration cell-type corrosion—250×.

"The abundance of the scale returned with the sample indicated that just such a condition exists. It is suggested that periodic flushing of the evaporator would serve to remove loose deposits of the type which accompanied the sample."

While prevention of corrosion from normal liquor, vapors or condensate is the primary design basis, serious consideration must be given to possible corrosion arising from the use of boilout liquors.

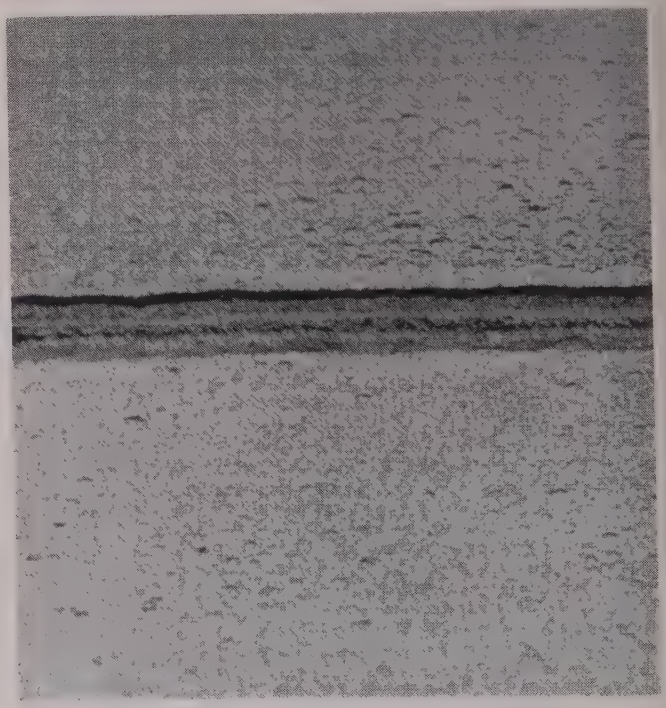

Fig. 9

Most evaporator operations sooner or later cause scaling of heating surface on the inside and/or outside surface of the tubes. The nature of such scale varies over a wide range of insoluble salts: silicates, oxides, sulphides, and organic material as well. As a consequence, a wide variety of boilout liquors has been used ranging from water to alkaline and acid solutions.

When employing these various boilout liquors, the tube material itself must be considered. Over the last number of years, many mills have gradually replaced carbon steel tubes with stainless steel, usually employing type 304 stainless of welded construction. Such tubes tend to stay cleaner for prolonged periods of time thereby justifying their extra cost in terms of prolonged high capacity and minimum lost production related to boilout down time and boilout cost.

Where a scale can be removed by water or alkaline boilout, there is little danger of damaging stainless tubes. However, some scales are not susceptible to such boilout procedure and stronger boilout chemicals are necessary. Inhibited hydrochloric acid has frequently been used for scale removal. A number of concerns are in the business of cleaning heat transfer equipment using various inhibited acid solvents. Great care must be exercised so that such acids do not lose their inhibited properties. 304 Stainless is highly susceptible to chloride attack. If the welded stainless tubing has not received a full finish including annealing and some cold working, serious corrosion due to chloride attack may result from hydrochloric acid boilout operations.

Figures 6, 7, 8, and 9 illustrate only too well a case of stainless steel tube failure from chloride attack where a series of inhibited hydrochloric acid boilouts attacked an inadequately finished set of welded stainless tubes.

Stainless tube manufacturers must be as much concerned with corrosion from boilout liquor as they are with normal operating liquors. The ASTM specifications governing welded tubing are much too broad. The buyer of such tubing should protect himself against inadequately finished welded tubing and should thoroughly discuss boilout as well as normal liquor requirements whenever buying a set of stainless tubes. Tube manufacturing procedures are being geared to these requirements.

Since stainless tubes represent a very high investment cost, every effort must be made to protect them. 304 Stainless will last a "lifetime" in normal kraft black liquor evaporators. They may be quickly ruined by improper boilout liquors particularly those containing hydrochloric acid unless such liquors are very carefully applied and unless the tubes are properly manufactured to begin with.

We recently made a survey of boilout practice in a number of mills and the findings are here summarized:

It is notable that the trend is definitely away from hydrochloric acid. In attempting to protect their stainless tubes,

"BOILOUT PRACTICE"—BLACK LIQUOR EVAPORATORS

Mill 1:	Water boilout—once per week Chemical boilout—twice per year, inhibited phosphoric
Mill 2:	Water boilout—once per week Chemical boilout—practically none during recent years
Mill 3:	Water boilout—once every week or 2 weeks Chemical boilout—rare, inhibited phosphoric
Mill 4:	Water boilout only—then soak hot machine with cold water
Mill 5:	Water boilout—once per month Chemical boilout—twice per year, inhibited hydrochloric acid
Mill 6:	Water boilout—as time permits Chemical boilout—Nalco phosphates
Mill 7:	Water boilout—scale so severe that frequent chemical boilouts required; used inhibited hydrochloric acid
Mill 8:	Water boilout—every 2 to 3 days Chemical boilout—once per year with Oakite "85"—recently tried 3% hydrochloric acid

many mills have directed their attention to the elimination of scale-forming materials. Recausticizing operations may have a direct bearing on the degree of evaporator scale. Liquor cleanliness and more frequent water boilouts may often produce a reduction in amount and type of scale such that mild boilout liquors or the water boilouts alone will maintain good cleanliness, particularly where stainless tubes are in use. Where carbon steel tubes are used, the introduction of amines into the vapor stream has given some indication of successful scale prevention. More work needs to be done to develop this application.

It is clear that 304 stainless steel, while meeting the major requirement of corrosion resistance to normal liquor operation, is susceptible to chemical boilout attack and is also susceptible to stress corrosion. Where failures have occurred due to stress corrosion, the cause is usually to be found in the method of tube rolling. Proper tube hole drilling is of equal importance with proper tube rolling. Tube holes must be drilled true and to the minimum tolerance compatible with tube diameter tolerance. Grinding of tube holes may cause subsequent overrolling. Automatic expanders which limit the torque applied on the roller are particularly important to stainless tubes. An overrolled stainless tube may fail at the tube sheet when subsequent operation produces vibration in the tube. Work hardening and stress corrosion go hand in hand.

A proper conclusion to this subject must, therefore, point up these sources of corrosion against which good design must guard: (1) corrosion due to normal liquor, (2) corrosion due to boilout liquor, (3) corrosion due to improper materials of construction, and (4) corrosion due to mechanical design.

Presented at the Thirteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, Portland, Ore., July 28–Aug. 1, 1958.

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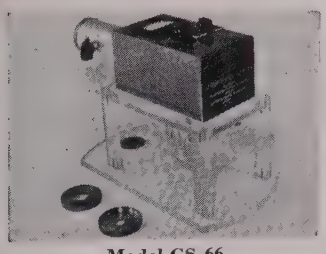

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The Effect of Machine Operation on Formation

HARRY E. HADLEY

THE title of this paper is in itself misleading inasmuch as formation means all things to all people, but I think it is generally agreed by all that when the wet sheet leaves the wet end and enters the drying section that nothing more can be done in regard to its formation; so therefore my remarks will be confined to the wet end or forming part of the machine itself. There is an old saying in the paperboard industry which states that "many machine tenders make poor sheets from good stock, but very few machine tenders can make a reasonably good sheet from poor stock"; so therefore in my remarks I will assume that the stock prepared for the machine is satisfactory for its intended end use.

Probably there is no place in the paperboard industry where the ability of the operating personnel will come forth more quickly than at the wet end of the board machine itself. Since all cylinder machines are built to operate over a very wide range of calipers, and handle a very wide range of types of fibers and form them into a sheet of board made up of multiple plys, it can be readily understood that there is a compromise built into the machine from the start, inasmuch as no machine could operate at its highest efficiency both in regard to production and quality in all the areas expected of it. Since this is true, then machine operation itself becomes very important to the formation of the sheet.

Since formation on the wet end is dependent on the proper application and handling of large amounts of water, then we should give some thought to this phase. We cannot discuss this in general terms because there are generally various types of water used, such as fresh water, white water (which may or may not contain alum or other chemicals), and what is generally termed as "dirty water," which may be water extracted from the filler cylinders, or a mixture of all the other waters.

It is therefore necessary that the machine operators bear in mind all the time that there are variables present which he must consider. There are few mechanical adjustments available to the machine operator; therefore the thoughtful use of the ones he has and the judicious use of the water quickly points out the good papermaker. However, there are some parts of the equipment over which he has direct control and is more aware of the condition of them than anyone else since he is closest to them.

Formation might be defined as the removal of water from a mixture of fiber and water. Let us consider briefly the factors which influence the removal of this water. We must consider this on the basis that we are going to handle the water in such a manner that the sheet being manufactured will serve its purpose and meet all of the specifications required of it in all respects, both in regard to physical strength characteristics and its ability to reproduce fine screen reproductions if necessary.

It has been my belief for many years that we operate in three areas in regard to formation difficulties, and not in two as is generally thought. By this, I mean that nothing goes from good to bad instantaneously generally, but has established a trend in one direction or the other and is headed that way for a period of time before it becomes visibly apparent. The good machine operator is aware of this fact and is continually alert to these trends. This can be illustrated in the following manner, and I am sure that we have all experienced this same thing or something very similar to it. When something happens on the machine that makes the sheet being produced unsatisfactory, we have often asked the machine tender what happened, and he readily states, "I don't know. It was all right a minute ago." That is generally not true except in cases of mechanical failure or breakdown; but the truth is that this condition has been developing for a period of time and has now become visibly apparent. The good operator, however, is aware of these trends and takes steps to correct them before damage is done. I have personally seen many times machines that have been running at apparent quality levels, and suddenly blows will appear either at the wet end or somewhere else on the machine, and investigation will immediately bring out that the sheet is crushing on one or more of the cylinders. This did not happen instantaneously by any means, but was developing for a period of time and not yet visible. However, good machine operators can detect this condition arising by close observation of his cylinders and by being aware of the conditions under which they are operating.

Probably one of the most important things that will contribute to good formation on a cylinder machine is good clean cylinders. If the cylinder faces are kept clean so that water can be handled through them, then we are going to get a uniform mat formed on their surfaces. However, when the opposite is true and the cylinder faces are allowed to fill up, investigation will generally show that the cylinders did not fill up uniformly all over their surfaces. They started to fill in some particular area and gradually developed a condition that became apparent to the operating people, at which time they had to take steps to correct. The ability of the cylinder wire to pick up stock is entirely related to the wire's ability to drain water. It is therefore possible to have a cylinder running that picks up more stock in some particular area than in others. This, however, is not conducive of good formation and will cause trouble.

The overhang and weighting of couch rolls is a very important factor. This again influences the removal of the water from the wet mass. Generally, couch rolls run in a fixed position on the machine; that is, they do not have adjustments readily accessible to the machine operator, especially in regard to overhang. This, in itself, can cause some difficulty at times due to the fact that, as stated above, the machines are expected to operate efficiently through a very wide range of calipers. The machine operator, however, does have a control over the weighting or the reduction of weighting of these rolls. In too many cases increased or decreased weighting is used to correct a condition that has nothing to do with the couching operation itself, but is due to the failure of the cylinders to operate properly. You have all seen couch rolls which have been jacked up in order to remove blows from following cylinders, or to cause the felt to pick the sheet off the cylinder face. This, of course, is good practice in cases of emergency but does not solve the basic operational deficiency, which of course should be corrected.

The handling and care of the wet end felts is a direct responsibility of the machine operators. Since no two machines are alike, the design of the felts used should be given careful thought so that they operate efficiently on that particular machine. We have five cylinder board machines in our plants, and we do not expect the same felt design to operate efficiently on all of them. I am sure that is true in any other multimachine mill. The felts are used on the board machines for three distinct reasons. In the case of the bottom felt, the first function it must perform is to pick the sheet from the cylinders and transport it through its continuing water removal operations. It also must have the ability to handle water; that is, to take it up and give it up rapidly and uniformly across the machine. On the forming section of the wet end, no water can be removed from the sheet that the felts will not take, so therefore the care of these felts becomes very important. We all know that to operate at its top efficiency the felts would have to run with the seams straight across the machine. Although this is done in many cases, it is general practice to run the felts with one edge lagging behind the other slightly in order to allow for stripping in case damage occurs to one on the edges of the felt while it is running.

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The third function the felt is to perform is to impart finish to the sheet. However, I actually believe that this would more accurately be stated that the felt should not distort the surface of the sheet any more than is absolutely necessary. When the felts are designed for the particular machine and are operated properly, they will serve all three of these functions.

As in the case of the cylinders, when a felt starts to fill up it reduces its water handling capacity and immediately starts to affect the formation of the sheet by distorting it in the press roll operation. This is the case again of things not going from good to bad instantaneously. We have all seen cases of where machine tenders coming on shift will say the felt is dirty and is on the verge of picking or crushing. I am sure that this did not happen just as the shifts changed but was developing over a considerable period of time. It is a case again of keeping the felts in good operating condition and not allowing them to develop inefficiencies to the point where corrective measures must be taken immediately or else catastrophe results.

The press rolls on the machine should be kept in good condition to maintain their desired contour so that they may perform the function they were installed to do, and which again was to remove the water from the wet mass of the sheet. Too many times press rolls are allowed to deteriorate in their ability to uniformly remove water to the point where additional load is put on other press rolls in the same machine to make up for the failure of one set of rolls which should be removed and ground or recovered if necessary.

It is my belief that all the equipment discussed here was put on the machine to contribute to the successful operation of the machine and to produce a uniform sheet. Therefore, it follows that if any one of these things fails to perform the function for which it was intended, or in any way hampers the ability of some other part of the equipment to perform properly, then it must be removed or corrected. It is always desirable to get as much wear from any part of the equipment as possible as long as efficient operation remains. However, sometimes to get 100% wear from any part of the equipment, or to achieve a few extra running days on a bottom felt, is the most expensive thing you can do.

The above is of necessity a generalization of machine operation, and I would like to sum up with a thought in regard to it. I have a definite feeling that the lack of communications between management and the machine operating personnel is a very important factor in this matter. This is a two-way street. Though management has a direct responsibility to communicate with the operating people, the operating people also have a responsibility to communicate with management in regard to the condition of the equipment. They are the best men to do this since they are the closest ones to it. I feel that if some brief educational program could be brought forth for operating people in regard to some of the simple laws of hydraulics, they would become much more proficient handlers of water. We have too many people operating machines who know that if they do certain things other things happen, but they do not have the foggiest notion of why they occurred. I think we should tell our operating people as many facts as we can in regard to the machine operation itself, and also keep them informed in regard to the sheet they are manufacturing. In many cases, they do not have any idea of what the sheet is going to be used for and what it is intended to do. They sometimes feel that specifications are merely put on orders to torment them. I am sure that if we would communicate these facts to the machine crews, we would then achieve some of the results we are striving for at all times.

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An Improved Measure of Dirt in Pulp and Paper

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Over the years the numerical measurement of the cleanliness of paper or pulp has been difficult, uncertain, and tedious. It is hoped that this contribution will serve to clarify the problem and possibly innovate an easier and more reliable testing procedure. Certain points which are generally, but not always adequately recognized, deserve some discussion:

Single Number Results

First, it is clearly desirable to have any test expressed as a single number. A dirt test is made to answer the question "how clean is this material?" To reply with a table showing the numbers of various sizes of specks found in it may be satisfactory to an analyst but is most frustrating to the user or buyer who is called upon to decide whether this or that material is cleaner, and by how much.

"Large" and "Small" Dirt

Second, to estimate and report the number of specks, even on the basis of "large," "medium," and "small" categories, is misleading, since one "large" black lump of dirt in a specimen of pulp, having a projected area of say 4 sq. mm., could be broken into 8 "medium" sized pieces of 1 sq. mm. area and these into 256 "small" pieces of 0.1 sq. mm. area. Thus, unless the specks are counted in many categories of sizes, very substantial errors in judgment could arise, particularly between products of different makes containing different kinds of dirt.

Color of Dirt

Third, a piece of black dirt in a sample of pulp, cut up and incorporated into a white paper, would make a far "dirtier" product than if it were the same sized piece of pale brown bark. Also, if the pulp containing the latter were made into an orange-colored paper, the brown bark would be far less noticeable than if the same pulp were made into white paper. Indeed, white specks (for example, from the dried foam from a previous furnish for a white paper) are objectionable and constitute "dirt" in a deeply colored paper. It is therefore necessary to take account of the contrasting color between the dirt specks and their background to get a true measure of their "dirtiness."

Numbering Small Specks

Fourth, the smaller the specks, the more there are, and the more tedious it is to count them on a large area. Furthermore, factors such as the color intensity and direction of the lighting, the distance of the specimen from the observer, the angle of viewing, his acuity of vision and his fatigue at making dozens of decisions as to whether this or that speck is large enough to be included in the count, make it almost impossible to expect concordance of test results obtained by a number count. Since it is often the scarcity of these smaller specks that make a paper appear "clean," the accuracy of their count is important.

Equivalent Black Area

Fifth, it is much easier to measure the "equivalent black area" (e.b.a.) of a speck than its true actual area (a.a.) unless the speck is round, has a standard black color, and is on a standard white background (when the e.b.a. and the a.a. are the same). The e.b.a. of a speck of dirt is defined as that size of black round spot on the TAPPI Standard Dirt Estimation

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Chart which, when placed adjacent to the speck, disappears (i.e., becomes indistinguishable from its background) at the same time as both are moved away together from the eyes of the observer.

A comparison of a speck with the reference spots is facilitated by throwing the eyes out of focus, by removing one's spectacles (if one has only a moderate acuity of vision), or else by examining both the speck and the spots together with a reading-glass held suitably out of focus. In any case, it should be clear that by these means, the e.b.a. of a speck of any size, color, or shape, on any surface can be estimated quickly, accurately, and objectively by simple comparison with the series of spots on the standard black and white chart. On the other hand, one cannot accurately measure the a.a. of a speck without using a comparison chart which has not only the same size, but also approximately the *same shape* as the speck. Even then, if other than black, care is needed (especially in the case of a speck having a low contrast with its background) not to underestimate its true area. The use of a transparent chart for measuring the a.a. is not any more helpful; it merely accentuates the difficulty of measuring the a.a. of a speck having little contrast with its background. By definition, it is not possible to use a transparent dirt chart to measure the e.b.a. of specks of various sizes, shapes and colors since not only these, but also the contrast between the speck on its background, and the reference spot on its standard white background (the TAPPI chart) are equally important factors.

Effect of Shape of Spots

Sixth, if one examines an actual TAPPI Standard Dirt Estimation Chart, it will be noticed that none of the rectangles stand out nearly so well as do the round dots having the same area. In fact, if examined with a reading-glass held out of focus, with the particular shape of rectangles used in that chart, it will be seen that their e.b.a. is only about two-thirds that of the round dots of the same actual area. (Incidently, in the actual photographic chart, the rectangles adjacent the 0.02 and 0.03 sq. mm. dots are invisible because of their thinness in relation to the resolving power of the photographic emulsions used.) If the rectangles shown were made still more slender, e.g., like long thin black fibers, and their a.a.'s were correctly depicted, the discrepancy between their e.b.a.'s and a.a.'s would be still greater. Thus, as a measure of "dirt"—apart from color and background, the true criterion is not the actual area of the dirt specks but their equivalent black area.

Surface and Total Dirt

Seventh, in the case of pulp, it has been experimentally established (*1*) as will be seen from Table I, that there is a close correlation between the visible dirt on the surface of a sheet of pulp and the total amount in the entire sheet of pulp, determined by converting it into thin sheets of paper (20 g. per sq. m.) and examining these. In making laboratory test sheets from a sample of pulp, it is most difficult to ensure that adventitious dirt is not picked up and included. Consequently, it is believed that it is neither necessary to count all the dirt specks in a sheet of pulp by wetting it and using transmitted light, nor first to convert the pulp into thin test sheets of paper for that purpose, with the attendent difficulties and danger of contamination. It will also be evident, that if a thick wet sheet of pulp is examined by transmitted light, it is impossible to estimate accurately the a.a. of the specks or their color, or accurately to establish a lower limit beyond which specks are then considered to be too small to be included in the count.

Reference Dirt Charts

During the last few years TAPPI methods T 213 m-43 and T 237 m-43 have had their effectiveness and standardization compromised by the issuance and use of improper charts, and especially by the recommendation (*2*) that a transparent chart be used to measure the e.b.a., which, as we have seen, is not possible.

Table I. Relative Estimate of Dirt on, and in, Pulp Sheets (*1*)

Pulp		E.b.a. of dirt on surface of pulp sheets, p.p.m.	E.b.a. of dirt in thin handsheets, p.p.m.
Bleached sulphite	K	3.3	3.0
Bleached sulphite	L	6.2	4.0
Another sample	L	6.2	4.5
Bleached sulphite	M	7.1	3.8
Unbleached sulphite	N	10.2	6.0
Another sample	N	11.0	8.0
Groundwood	O	11.2	6.8
Unbleached sulphite	P	12.0	7.7
Unbleached sulphite	Q	12.8	7.0
Another sample	Q	14.7	7.1
Unbleached sulphite	R	23.0	10.5
Another sample	R	24.0	10.4

In the printing of the original (large) 1933 chart (*3*) the ink spread and enlarged the smaller spots very substantially, for example, the 0.05 sq. mm. spot spread to 0.08 sq. mm. Unfortunately, this same error was compounded about 1954 by the preparation and distribution of large transparent charts copied from a 1933 printed chart. In 1955, still other charts were made by coating standard TAPPI postcard-size charts with plastic, so as to keep them clean. If the spots on the plastic-coated chart are compared with those on the uncoated standard TAPPI chart, it will be found that the e.b.a.'s of their smaller spots are considerably less than standard, by as much as 25%, so that if the plastic-covered charts are used, their designated areas should be corrected—a matter which depends largely on the lighting, the angle of viewing, and the size of each spot.

The e.b.a.'s of the circular spots on the uncoated postcard-size photographs are quite accurate. In accordance with Graff's findings (*4*) all the spots on this chart, except the following, are correct within 2% or 0.005 sq.mm. (whatever is the larger). For especial accuracy, the designated areas in square millimeters of the following spots should be changed to those given in parentheses: 1.00 (1.08), 0.80 (0.76), 0.60 (0.58), 0.40 (0.42), 0.30 (0.31), 0.25 (0.26), 0.20 (0.21), 0.15 (0.16), 0.10 (0.11), 0.9 (0.10).

Specimen Size and Speed of Testing

Drawbacks to the wider use of T 213 m-43 and T 437 m-43 have been, first, their tediousness to perform and, second, the specimen size generally is not adequate to be sure about the representative inclusion of the larger spots present. In evolving those two methods (*3*) the sizes required for the specimen were held to a minimum so as not to prolong the test unduly. Recently, by a modification to the procedure, which will now be described, both these drawbacks have been overcome to a great extent.

The smaller dirt specks in pulp or paper probably are more numerous in inverse proportion to about the square of their size. Thus to secure a representative estimate of their presence it is necessary to examine only a correspondingly smaller area of the specimen sheets. Furthermore, the larger specks are more easily seen, and it is not difficult to scan a relatively large area of the pulp or paper sample for their presence. Accordingly, a considerable saving in time and increase in accuracy can be gained as follows.

New Procedure

Select, so as to be representative of the shipment, at least 20 specimen sheets, each having an area of not less than 500 sq. cm., or in the case of pulp, at least twice this area. (This is five to ten times the area previously specified.) Examine the

Table II. Dirt Estimate for a Medium Grade of White Bond

Two specimen lots A and B, each 20 $8^1/_2 \times 11$ in. sheets. E.b.a. of dirt specks in hundredths of square millimeters

Sheet no. Lot A	Felt side Large	Felt side Small	Wire side Large	Wire side Small	Sheet no. Lot B	Felt side Large	Felt side Small	Wire side Large	Wire side Small
1	...	4	..	4	1	...	5	..	..
2	...	5	7	5	2	7	4	..	4
	7								
3	18	..	..	3	3	...	..	..	..
							6		
4	...	6			4	13	5	8	5
	8								
5	22	4	8	4	5	20	..	..	..
6	...	..	..		6	...	5	10	..
			15						
7	...	4	15	5	7	8	..	..	6
8	7	4	..		8	...	..	..	4
9	7	..	..	6	9	...	4	..	..
				4					
10	...	5	15	..	10	8	..	20	6
						11			
11	8	..	9	..	11		..	..	4
12	7	4	..	5	12	20	..	7	..
13	...	..	..	..	13	...	6		4
14	...	4	..	..	14	15	..	8	..
15	...	4	..	6	15	...	..	..	..
16	...		8	5	16	...	4	9	6
								11	4
17	...	6	10	6	17	28	..	..	..
		4							
18	7	..	12	..	18	...	4	20	..
	22						5		
19	...	6	..	..	19	...	..	..	..
20	7	..	..	..	20	...	4	..	5
Sum	120	60	99	53	Sum	130	52	93	48

Area specimen sheet = $8^1/_2 \times 11$ in. = 94.5×6.45 = 610 sq. cm.

Lot A Dirt felt side = $120 + (4 \times 60)$ = 3.60 sq. mm. $\times \frac{1{,}000{,}000}{610 \times 20 \times 100}$ = 2.95 p.p.m.

Dirt wire side = $99 + (4 \times 53)$ = 3.11 sq. mm. $\times \frac{1{,}000{,}000}{610 \times 20 \times 100}$ = 2.55 p.p.m.

Average dirt for lot A 2.75 p.p.m.

Lot B Dirt felt side = $130 + (4 \times 52)$ = 3.38 sq. mm. $\times \frac{1{,}000{,}000}{610 \times 20 \times 100}$ = 2.77 p.p.m.

Dirt wire side = $93 + (4 \times 48)$ = 2.85 sq. mm. $\times \frac{1{,}000{,}000}{610 \times 20 \times 100}$ = 2.33 p.p.m.

Average dirt for lot B 2.55 p.p.m.

Grand average for both sides of paper 2.65 p.p.m.

specimen sheets and select a particular spot on the Standard TAPPI chart that has an e.b.a. such that there will be, on the average, at least one such sized speck or larger, to be found on each 1000 sq. cm. of the exposed surfaces of the specimens. (The size of the particular spot selected might vary from 0.06 of a sq. mm. for a very clean writing paper to 0.25 or more for a dirty paper or pulp.) From an extra sample sheet, prepare a mask the same width as a specimen sheet, but three quarters its length and, if the pulp in paper is very dirty, another mask with one sixteenth of its area, exposed, by cutting away, for example, a rectangle at its top right hand corner. The portions exposed may be changed to one-fifth and one-twentieth to facilitate the subsequent calculation.

Number each specimen sheet, preferably on the top right-hand corner of its felt side. Pick up a specimen with one hand (if thin or transparent lay it on a stiff white backing) and hold an edge of the TAPPI Standard Dirt Chart against it with the other. Ignore the small specks less than the size of the selected spot but examine all the larger dirt specks on the felt side of the specimen, and, after scratching each speck with a corner of the chart to make sure that it is not a piece of loose, outside dirt, estimate and record its e.b.a. if equal to or greater than the e.b.a. of the particular reference spot previously selected. When the e.b.a.'s of all the large specks have been recorded, place the three quarter mask on the lower part of the specimen sheet and examine the exposed quarter area at the top for specks smaller than the larger reference spot (but not less than 0.04 sq. mm.) and record these. Turn the specimen sheet over and examine and separately record the larger specks on the entire surface, and the smaller specks on the lesser surface of its wire side, in the same way.

If the sheet is very dirty, select two particular sizes of reference spots—a "big" and a "medium" spot of about half the size of the larger, such that at least 10 "big" specks will be found on the 20 sides of the entire specimen sheets and at least 10 "medium" specks on one quarter of this area. After estimating and recording all the "big" and "medium" specks on the face of a specimen, the latter with a three quarter mask, use another mask exposing only one sixteenth of the sides of the sheets to estimate the e.b.a.'s of those specks less than the e.b.a. of the "medium" spot down to 0.04 sq. mm.

During the count, if an odd piece of dirt is encountered which is obviously unusual, e.g., a crushed insect, or a blotch of dirt which is not representative of the shipment (ascertained by looking over additional sheets), ignore it.

Shives or specks which appear dark only at some particular angle of observation are not counted as dirt. However, if their number is noticeable, they should be recorded separately in the report, giving their approximate actual average area, their color or appearance, and the average number per square meter, on each side of the specimen.

If there are an unusual number of specks present in the sheet less than 0.04 sq. mm. and over 0.01 sq. mm. which normally are too small to be regarded as "dirt," their total e.b.a. may be estimated by scanning one sixteenth or less of the area of the specimens and reporting their total e.b.a. separately.

Measure the area of the specimen sheets. Calculate the total e.b.a. of dirt on the wire and felt sides of the sheets separately, by totaling the e.b.a.'s of the larger specks on the entire area and adding four times the e.b.a.'s of the smaller specks observed if the three quarter mask was used, in accordance with the lesser area examined.

Report the results as square millimeter of equivalent black area of dirt per square meter of surface examined, i.e., in parts per million, for the wire and for the felt sides of the paper, or pulp sheets also their average, noting the presence of any unusual number of shives or tiny specks as described above.

Table II gives an example of the new procedure as applied in duplicate to a white, medium clean, bond paper, using two

separate lots of twenty $8^1/_2 \times 11$ in. specimen sheets. Each determination took about 20 minutes. In the table, the e.b.a. of each speck is recorded in terms of hundredths of a sq. mm. so that specks having an e.b.a. of less than "4" are normally not counted. The total area of the specimens examined in this example is six times the minimum area called for in T 437 m-43. It is believed that although less time was consumed in making the test, the results are considerably more reliable.

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Advanced Experimental Design in Cellulose Experimentation

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In spite of a great deal of interest in statistical methods, few applications appear. Part of this difficulty may result from the fact that introductory statistics do not consider problems of primary use to the physical scientist or engineer. In the following discussion, this contrast is defined, and several useful concepts such as sensitivity and efficiency are considered. An example of a relatively sophisticated problem in design and interpretation concludes the discussion.

STATISTICAL METHODS FOR DISCONTINUOUS VARIABLES

Until recently, statisticians have been primarily concerned with experimental designs involving the comparison of treatments. Treatments such as type of reagent or variety of wood are discontinuous variables.

The problem in experimental design in this case is to make accurate comparisons of a relatively large number of treatments under the restriction that only a few treatments can be compared under uniform conditions. For example, compare ten varieties of wheat when the size of a field with uniform soil characteristics will allow only three varieties of wheat in one field. As a second example, compare five types of bleaching procedures when the amount of material from a single digester charge is only large enough for two bleach charges.

The problem of analysis of data from these designs is to sort out genuine effects from apparent effects which can be as readily attributed to experimental error. The technique of variance analysis is the most frequently used statistical tool. The procedure is generically that of significance testing and the question that of avoiding type I errors, i.e., claiming to have found an effect where none in truth exists.

STATISTICAL METHODS FOR CONTINUOUS VARIABLES

In pulping and other cellulose experimental work, the practical problem is usually quite different. Here are variables such as pressure and concentration which are continuous in contrast to the discontinuous variables of the preceding section.

The problem in experimental design is also different. Generally, the relationship of variables is of interest. The questions are concerned with the form of the relationship. The search is for designs that will be efficient in estimating a relationship. For example, determine the relationship between active alkali and unbleached yield or between sulphidity and pentosan content or between pH in the sour and calcium content.

A closely related problem to that of estimating the relationship is to prepare a program that is sensitive in detecting the influence of variables.

The statistical techniques are those of estimation, e.g., least squares. It is now principally a problem to avoid type II errors, i.e., failure to find a relationship that truly exists.

In the remainder of this discussion, several of the above concepts will be briefly discussed. The rigorous arguments or extensive experience which could be given in each case in support of the conclusions will be omitted.

DETERMINING THE FORM OF A RELATIONSHIP

Ideally, a theoretical relationship can be written for the variables in question, and the experimental design problem is then to estimate in the "best" manner the unknown parameters. In alkaline pulping, it is necessary to be somewhat more practical.

In our experience, a simple empirical relationship is adequate in all cases thus far encountered. For example, the practical ranges of temperature, active alkali, and sulphidity are not great. The problem is simply to find out how the variables are interrelated in their influence on yield or purity so that one can interpolate to a useful set of cooking conditions.

Specifically, quite often a simple linear relationship is adequate, and in all other cases a quadratic or second-order relationship is sufficient.

The magnitude of experimental error in cellulose work is such that more elaborate relationships than the above have yet to be justified in a practical example.

In some cases, of course, it is known that the logarithm of time or some other simple transformation of a variable is appropriate. Certainly it is inexcusable to ignore such information in designing an experiment.

There is some emphasis in statistical circles on elaborate designs (highly fractionated factorials) in which a large number of variables can be screened. It is our experience that this technique is not usually required. Any good cellulose chemist can usually narrow down to a half dozen or less the really important variables in a system.

Finally, it is important to remain aware that the selection of variables influences the apparent behavior of a system. Expressing concentrations on a solution basis or on a moisture-free wood basis may well give quite different relationship among the variables. Since empirical description and interpolation is the principal concern, it is wise to select the variables to give as simple a relationship as possible. Some trial and error and experience is needed here.

EFFICIENCY IN DESIGN

Consider a simple problem in relating unbleached alpha-cellulose to active alkali concentration in the cook. How many levels or different concentrations of active alkali should be used.

Suppose we can afford to do 12 experimental cooks. Suppose also we can assume a linear relationship in the region under study. There are then several possibilities ranging from doing six cooks at each end of the range of active alkali of interest to doing 12 cooks at equal intervals throughout the range. It can be shown that the efficiency of the second alternative is about one third that of the first.

More realistically than in the preceding paragraph, some curvature can be expected, and it is then most efficient to do four cooks at each end of the range and four in the center of the range.

It is sometimes felt that scattering the experiments through-

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out the range of interest will prevent the engineer from missing something.

In reference to the previous assumptions concerning empirical relationships, 12 experimental locations throughout the range of interest implies directly that an eleventh order relationship is of interest, which is of course absurd.

As a general rule, the number of experimental locations should be kept to a minimum consistent with assumptions made about the type of relationship.

SENSITIVITY IN DESIGN

Although efficiency in design requires the use of the minimum number of experimental locations, it is still necessary to consider the number of experiments to be carried out at each location.

The erroneous impression is too often given that sophisticated experimental designs allow the experimenter to get by with fewer experiments.

In systems where experimental error is negligible with respect to the effects produced by the variable, it is true that a sophisticated design because of its greater efficiency may permit a smaller number of experiments to give the same sensitivity. In cellulose work, the experimental error is often of the same order of magnitude as the deliberately produced variation in the system. In these circumstances, a certain amount of replication or repeated runs at each experimental location will be required even with a design of maximum efficiency.

For instance, consider the example of unbleached alpha-cellulose and active alkali in the cook. In accordance with the recommendations of the previous section, select three levels of active alkali for the experimental locations. The number of experiments at each location is then determined by the sensitivity desired. By this is meant that when the coefficient of the linear term in the relationship is calculated from the data it should be significantly different from zero if the true value is different from zero by a certain number of standard deviations. In the present case, if three experiments are done at each location, the linear coefficient will be significant if the true value is about 1.8 standard deviations; if nine experiments are done at each location, then the linear coefficient will appear significant if the true value is 0.8 standard deviation in magnitude.

TYPES OF EXPERIMENTAL DESIGN

With the above concepts of sensitivity and efficiency in mind, the experimenter is better prepared to consult the statistician in the use of sophisticated designs. Textbooks such as Davies, "Design and Analysis of Industrial Experiments," will furnish all needed information. It should be pointed out that many standard texts are not written by people familiar with the problems of experimentation in physical or engineering science. Generally, most books written before 1953 are inadequate, and many books since 1953 are not much better. The reason for this is that statisticians have only very recently come to recognize and treat adequately the problem of experiments with continuous variables.

USE OF PLANT RECORDS

Because of the emphasis on empirical relationships and the availability of electronic computers, considerable interest is currently directed toward the use of plant records for determining relationships.

This procedure has several disadvantages if not actual dangers. If data from a plant log are plotted, most of the information will be found in a central area about the normal operating point and very little information at any distance from normal conditions. Under these circumstances, the data in the normal operating region provide almost no information about linear slopes, and the deviant points (those most suspect from an operating standpoint) are weighted by a very sizable amount. The better the past operations have been the less satisfactory the data will be for determining relationships.

Even if an equation is derived by manipulating historical data, it can never be expected to be anything more than a description of past experience, and its use for prediction is extremely risky.

On the other hand, a relationship derived from a properly designed experiment is more than a description of the data and can reasonably be expected to have predictive value.

DISCUSSION OF AN EXAMPLE

In the appendix is presented an example of a moderately sophisticated experimental design application. The example can be considered to illustrate points in the above discussion as well as offering some additional material on handling the computations that are involved with this technique.

SUMMARY

It has been pointed out that the concepts and techniques in the field of experimental design that are most applicable to problems involving continuous variables are not ordinarily encountered in introductory considerations of statistical methods.

Stripped of mathematical discussion, some of these concepts have been reviewed in order to permit the alert experimenter to make better use of statistical knowledge available to him.

A practical example serves to illustrate the possibilities of recent developments in the field of experimental design.

APPENDIX

The following experiment was conceived and carried out by Wendell L. Thornburg of the Research Department.

The problem was to determine the influence of temperature, sulphidity, chemical concentration, active alkali, and time in the sulphate cook on a special wood preparation.

A second-order composite design in five variables was proposed. The single replicate of 27 experiments is sufficient to detect linear coefficients of the magnitude of one standard deviation.

Levels of Factors

Factor	*Level*				
Code	−1.547	−1.0	0	1.0	1.547
Temp., °C.	149	165	195	225	241
Time, hr.	0.4	0.65	1.3	2.6	4.0
Sulphidity, %	1.8	10	25	40	48.2
Conc., g.p.l.	24.6	30	40	50	55.4
Active alkali, %	12.3	15	20	25	27.7

Note that the time variable is actually log t. The experimental runs were carried out according to the following plan but in random order.

Experimental Design[a]

Run No.		*Run No.*	
1	[a]	15	a c d e
2	a b	16	b c d e
3	a c	17	0
4	b c	18	$+\alpha$A
5	a d	19	$-\alpha$A
6	b d	20	$+\alpha$B
7	c d	21	$-\alpha$B
8	a b c d	22	$+\alpha$C
9	a e	23	$-\alpha$C
10	b e	24	$+\alpha$D
11	c e	25	$-\alpha$D
12	a b c e	26	$+\alpha$E
13	d e	27	$-\alpha$E
14	a b d e		

[a] The letters are not keyed to the variables in the preceding table.

The first 16 experiments are a one-half replicate of a 25 design in the usual notation where the presence of a letter designates the +1 coded level and the absence of a letter designates the −1 coded level. Experiment no. 17 is the center of the design and the remaining runs the axially symmetric locations where all factors are at the O coded level except the one designated. The selection of $\pm\alpha = \pm 1.547$ results in an orthogonal design.

Data were collected on each experimental run for several parameters.

The orthogonal coefficients for each term are directly developed from the coordinates of the experimental locations.

Orthogonal Coefficients[c]

K_A	K_{A^2}[a]	K_B	K_{AB}[b]
−1	0.23	−1	+1
−1	0.23	−1	+1
−1	0.23	−1	+1
−1	0.23	−1	+1
−1	0.23	+1	−1
−1	0.23	+1	−1
−1	0.23	+1	−1
−1	0.23	+1	−1
+1	0.23	−1	−1
+1	0.23	−1	−1
+1	0.23	−1	−1
+1	0.23	−1	−1
+1	0.23	+1	+1
+1	0.23	+1	+1
+1	0.23	+1	+1
+1	0.23	+1	+1
0	−0.77	0	0
+1.547	1.62	0	0
−1.547	1.62	0	0
0	−0.77	+1.547	0
0	−0.77	−1.547	0
0	−0.77	0	0
0	−0.77	0	0
0	−0.77	0	0
0	−0.77	0	0
0	−0.77	0	0
0	−0.77	0	0

[a] $K_{A^2} = K_{A^2} - K_{A^2}$.
[b] $K_{AB} = K_A K_B$.
[c] This table is not keyed to the experimental design table.

The coefficients for the response equation in the orthogonal polynomial form are computed as $b = \Sigma Ky/\Sigma K^2$ and the standard deviations are σ_b^2, $\sigma^2/\Sigma K^2$.

The general equation is written as follows:

$$y - \bar{y} = b_A K_A + b_{A^2} K_{A^2} + b_{AB} K_{AB} + \ldots$$

The coefficients of the various terms are tested for significance against their standard errors. Practically speaking, the significant and nonsignificant terms are usually readily separable into two groups.

For the yield in the present experiment, three terms were significant: A, A^2, and AD. The equation was as follows:

$$y - 33.52 = 4.533K_A - 2.875K_{AD} - 3.794K_{A^2}$$

To plot contour maps of the response surface, it is desirable to rewrite the equation in terms of powers of K_A, K_B, etc., using the relationship in the footnotes of the table of orthogonal coefficients.

$$y - 33.52 = 4.533K_A - 2.875K_AK_D - 3.794(K_{A^2} - \overline{K_{A^2}})$$

$$y = 4.533K_A - 2.875K_AK_D - 3.794K_{A^2} + 36.441$$

For selected values of y (contour lines), the equation can be solved for values of K_A corresponding to selected values of K_D.

Although the equation is valid only within the area in which experimentation has been done, it is often desirable as in this case to plot over a larger area in order to visualize the surface more easily (see Fig. 1).

It is of course possible to transform the equation to the original variable, but it is in general much simpler to keep the coded form.

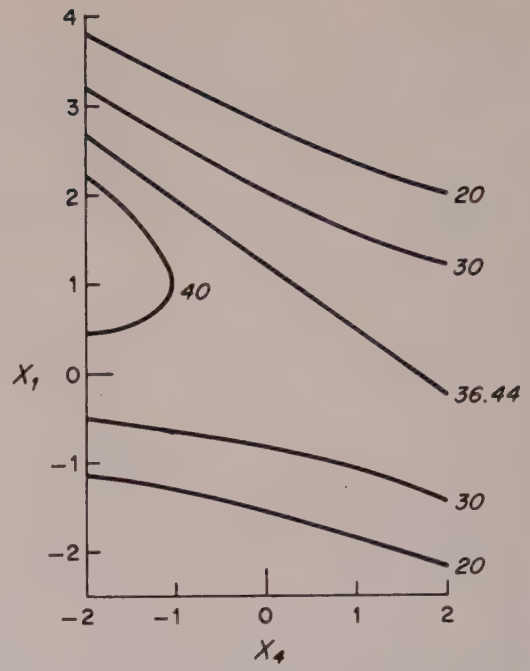

Fig. 1. Response surface, % yield = $f(X_1, X_4)$

The equation and the contour surfaces contain the estimated information from the experiment. It is unwise to make estimates at a particular point without some consideration of the variability or confidence limits for the estimate.

Two approaches are useful. First, it is possible to prepare from the equation a contour map of error of estimation, and secondly it is possible to compute the error at a particular estimated point in the factor space. Use of the original orthogonal form of the equation for computing error estimates is desirable as it avoids mistakes traceable to combining terms.

Two error estimates are of interest:

1. $\sigma_y^2 = \sigma^2\left[1 + \frac{1}{n} + \Sigma\frac{(K_i)^2}{\Sigma K_i^2}\right]$

2. $\sigma_y^2 = \sigma^2\left[\frac{1}{n} + \Sigma\frac{(K_i)^2}{\Sigma K_i^2}\right]$

The first is the variance of a single observation at a predicted location. The second is the variance in a predicted location due solely to errors in the empirical relationship. The summation is over all terms used in the equation at the coordinates of the predicted location. Note that σ^2 is aminimal error estimate for comparing an additional experiment with a predicted value and in practice is generally adequate for predicted values in the region in which the experiment is carried out.

In cellulose work, many dependent parameters are estimated for each experimental run. Since these parameters are not independent joint statements about several parameters made from the separate equations for each should be treated with reservation. Techniques for setting confidence limits on such statements are available.

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Machine Design and Cylinder Machine Formation

WM. S. MILNE

THE purpose of this paper is to explore some of the areas in which machine and equipment design affect formation and thus the finished sheet of multicylinder board. In order to explore these areas, agreement must be reached on a definition for the rather general term "formation." For the purpose of this paper, good formation will be defined as that fiber orientation at the moment of felting which produces the specified sheet at the dry end of the machine. As an appendix to this definition we will also consider that there are a number of things which can disrupt this sheet before it gets to the dry end but few, if any, that can improve it. In the comments presented, only those disruptions taking place while the sheet is on the mold or the making felt will be considered.

By the above definition, good formation varies with the use of the product made from the finished board. Obvious examples range from corrugating medium, where machine direction fiber alignment is needed to produce high compressive strength in the finished box, to milk bottle stock where a reasonably square sheet is wanted for a rigid carton. Other examples, which come readily to mind, are patent coated grades requiring a well closed sheet for efficient liner coverage and good finish and printability and gypsum board grades where uniform porosity is so important to insure proper binding in the later steps of the process.

Thus, it is that the finished boards' use dictates which of the components, such as squareness, finish, strength, pick, and porosity are needed to produce a well formed sheet. In addition to these properties, there is one quality of good formation that is universal. This quality is that of a truly level sheet, and refers to both uniform thickness and uniform weight. This quality is needed both for itself and for the effect it has on other steps in the producing and converting processes.

In order to combine the desired formation components in a sheet, it might be considered that there are four major variables in papermaking. These variables are the type of raw stock available, the flexibility of the stock preparation equipment, the skill of the papermaker, and the flexibility of the forming part and related equipment. Assuming the first item to be relatively constant, close attention to the design of the fourth item should allow a greater portion of the operators skill and the stock preparation systems' flexibility to be put to use improving the operation rather than making up for deficiencies of the forming part.

VAT SYSTEM DESIGN

One of the first components to consider in vat system design is the screen which may or may not be actually in the vat system. Side-stepping their location, screens and other cleaning equipment must provide a stock that is free of oversized and foreign particles if good formation and appearance are to be secured.

It should also be mentioned that pressure screens, such as the Selectifier, when properly installed in the vat system have proved useful as defloccing tools as well as screens.

Assuming adequate cleaning, the stock must be introduced into the vat system in a constant and controllable manner. Regardless of the type of distribution system installed, it seems desirable to include consistency control as an aid to consistent operation and good formation. The distribution system should be designed to eliminate all fluctuations other than the actual adjustment made by the operator. The stock should be introduced and mixed in a manner to avoid all possible air entrainment and the drainage and pump problems it causes.

Since a certain amount of entrained air seems to find its way into any vat system, it may be well to consider some means of removing it. Several recent installations have been made in which tanks or chests have been incorporated in the white water return. The purpose of these is to allow a retention time long enough for the water to give up its entrained air either by natural forces or by a vacuum drawn on top of the unit. Since these units require space, it would appear well to plan for them in any new installation.

In the same connection, care must be taken to pitch the vat system piping so that pockets for air bubble formation are avoided.

The vat system piping should also be designed with the type of system in mind, as well as the operating values of flows and consistencies over the entire expected range of grades to be made. Gravity fed pipes should be large enough so that flow is not restricted. Pump fed pipes should be sized to provide reasonable velocities so that stock build-up as well as excessive power costs are avoided. Piping should be arranged so that wash out procedures can be easy and effective.

In the selection of fan pumps it must be remembered that to provide the operator with dependable adjustment, excess pump capacity is required. As a rule picking a pump for maximum flow with the discharge valve only two-thirds open will give this control.

The vat system piping ends with the final connection to the vat. Originally this was done by simply flanging the pipe to a convenient spot on the vat. The early machines which were fed in this manner were narrow enough and ran few enough grades so that this type of connection could be made to work. As times and machines changed many elaborate and costly ideas have been applied in hopes of taking the stock from a round pipe and spreading it uniformly across the width of the machine. It is interesting to note, however, that the large bulk of new machines and new vats have been equipped with relatively simple hopper-type inlets. Such an

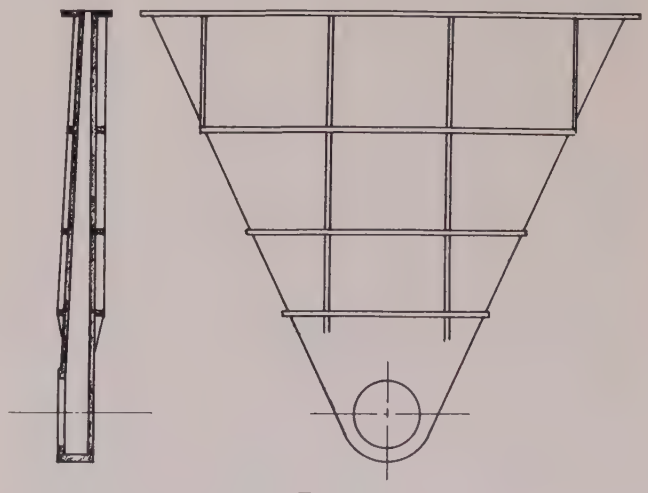

Fig. 1

inlet (Fig. 1) should be of double tapered construction so that the width of its slot narrows as the sides of the hopper fan out toward the full width of the machine. The entrance pipe to such an inlet appears to be best directed against the hopper wall at right angles to provide uniform turbulence in this area. Any such hopper must be adequately reinforced to withstand the pressures involved, so that the slot width does not vary due to distortion. As with other elements of the vat system, adequate provisions should be provided to clean out this or any other inlet.

It should be noted that the double tapered hopper described

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above appears to be adequate for today's machine speeds and widths. However, since the angle of fanning of this type of inlet is somewhat critical, there is probably a limit to the width of a machine which can be supplied by this means, using a reasonable basement depth. If we are to continue to

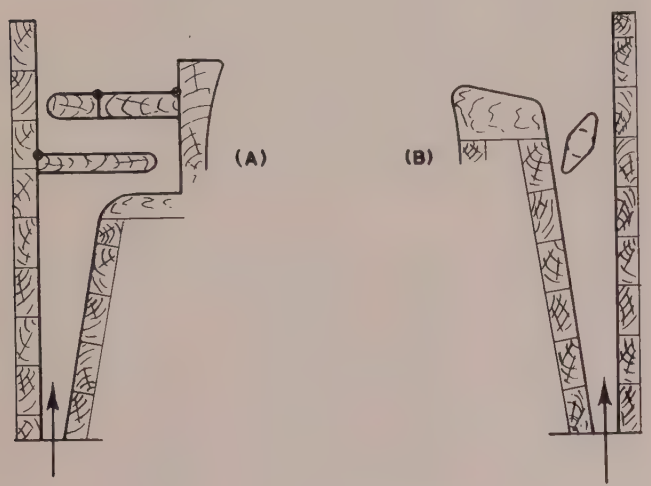

Fig. 2

increase the width of the cylinder machine with the idea of some day matching the width of today's wide fourdriniers, this will certainly be an area for new development and design.

VAT AND CYLINDER MOLD DESIGN

Progressing from the last item of the vat system, attention must be turned to the approach flow compartment. The present popular theory seems to be that the sooner the stock reaches the forming area after it is spread, the better. On this basis, most recent machines and vat additions have been equipped with short vertical approach flow compartments (Fig. 2A and B). On counterflow vats these passages have horizontal baffles which are hinged to provide easy access for wash-up. Based on some recent installations it may be possible that this type of baffling can be satisfactory on direct flow units as well.

In keeping with the comments on the reduced length of approach flow compartments, the actual taper of the inlet hopper has sometimes been carried up inside the vat ends fairly close to the first horizontal baffle. In addition to shortening up the approach flow compartment distance, this also assists in keeping the depth of the inlet hopper to a minimum.

For machines producing a wide variety of grades, the top baffle can be made with an adjustable end; this affording the operator control over the turbulence of the stock as it reaches the top of the inlet compartment.

As indicated above, there have been recent occasions where a baffled inlet, similar to that described for the modern counterflow vats, has been successfully applied to direct flow vats. Probably more conventional for the approach flow compartment of a Uni-flow vat (Fig. 2B), is a relatively simple fan shaped compartment, rising from the pinch point at the hopper connection. A single restriction similar to the adjustable top baffle in the counter flow unit is usually found in this arrangement. This restriction is called a butterfly valve and it provides the operator control over the orifice width in the approach compartment.

Distributor rolls are found in the approach flow to some recent vats and certainly deserve mention. These rolls have been used both submerged in the throat and only partially submerged on top of the making board. In the first location the principal reason for limited acceptance is probably simply a matter of cost for benefit received.

In the partly submerged location, a major problem is that of cleanliness of the roll itself since strings are apt to be formed. The showering of a roll in this location directly under the felt is awkward if not impossible.

Having successfully provided a uniform mixture of stock and water through the approach flow compartment to the making board, the verticle location of the cylinder mold in relation to the making board should be carefully considered. This relationship is important because it determines the angle of emergence of the mold for the various stock flows to the mold. This angle is measured by the angle between the horizontal and the tangent to the mold surface at the point it comes out of the suspension. The larger this angle, the more possibility for slippage and roll backs on free stocks. For counter-flow vats, there is little that can be done to change this other than changing stock conditions. On a direct flow vat, adjustment is normally provided by raising the overflow dam; but this adjustment is still limited by the location of the weir on the incoming side of the vat.

Once stock has been introduced over the making board into the vat circle it must still be kept in uniform suspension around the submerged circumference of the mold. For this reason clearances between the mold and the vat circle require considerable attention. In the past, designs have been proposed for vats with adjustable circles. This theoretically desirable design, in the majority of cases, is not used as an operating adjustment after initial start-up. Vats with adjustable circles are expensive. This probably partly accounts for the continued popularity of the wood making board and circle which can be altered if need be.

One additional control mechanism which should be mentioned and which requires consideration in the design of counterflow vats is the wing dam. These adjustable dams located on the making board provide the operator with a convenient means of correcting the sheet profile which is naturally thin on the ends.

In addition to the stock passages in the vat, those provided for white water require careful design. The vat ends themselves should be equipped with adequate wash-out provisions. Spill and white water compartments and connections must be of sufficient size. One advance recently made in this direction (Fig. 3) consists of locating the mold packing glands on the outer rather than the inner wall of the vat end casting. This affords absolute maximum area for white water removal out of the end of the mold. This change also makes it possible to provide air exhaust connections which are sized by engineering calculation rather than the space available on a conventional gland plate.

The air exhaust system is a common addition to modern vats removing air from within the mold. The resultant slight vacuum lessens "rain" between molds, foam inside the cylinders and slippage of the formed sheet as it emerges from the stock in the vat.

It is believed to be generally accepted that spill and white waters should be taken from both ends of each vat to minimize mold head gradient across the vat. A difference in the depth of white water at the ends of the mold because of spills

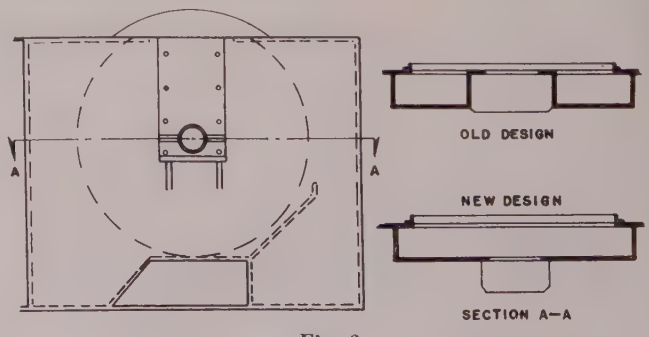

Fig. 3

on both ends is prevented by having the spill gates cross indexed.

Just as vat end design is of importance in providing adequate passages and controls for operation, so should cylinder mold design be reviewed. The first basic requirement is a purely mechanical one that the mold be rigid and round so that good wire life is secured, as well as stability in operation. Assuming the mold to be of conventional design with central core and spiders, the core size must be selected to suit the span since little or no strength can be counted on from the outer elements of rods and wire. In this connection the mold used with the vat ends described above has a core extending beyond the end spider into the vat end.

It has been mentioned earlier that one can expect entrained air in any vat system. A certain portion of this is introduced by the churning to the spider arms in the white water. To keep this churning to a minimum, these spokes should have a streamline cross section. The spiders should be mounted on the core so the arms are staggered and do not all strike the water at the same time.

Rods and winding wire shape should be considered for their effect on turbulence as they pass through the vat and as the white water passes through the mold face.

Face and foundation cloths as well as rods and wires should be chosen and installed so that they can be properly cleaned. On liner vats particularly this will probably mean a compromise between wire marking and a clean mold. On almost any vat an effective mold shower is a must.

PRIMARY WATER REMOVAL DESIGN

Assuming a well formed sheet is laid up on the mold as it emerges from the vat, the area is entered in which formation is disrupted but not truly made.

Not too many years ago the wet felt was required to do double duty by serving as a power transmission belt to drive the major portion of the wet end, as well as providing a means for couching the sheet off the mold and taking water in the presses. The picture has been materially changed by development of accurate helper drive equipment—to the extent that most modern wet ends are equipped with helpers on at least half of the cylinder molds and at least one roll in each of the primary water removal pairs. This change has allowed the felt manufacturers to concentrate on a felt weave which has better draining characteristics, leaves less of a mark on the sheet and remains open over a longer period of time—reducing crushing problems.

In connection with the above comments on helper and felt design, it must be remembered that effective cleaning of the felt must be provided in order to secure efficient operation over a reasonable life cycle. The time-proved method of cleaning such felts is a felt whipper and showers. The whipper design should be adequate for the high stresses involved. One of the rolls either directly before or directly after the whipper should be provided with an adjustable mounting so that the force of the blow struck by the whipper bat against the felt can be set at the best place for effective felt cleaning.

Returning to the point at which the sheet is couched by the felt from the mold, consideration must be given to the various design elements of the couch roll, including its mounting. The couch roll cover should probably have a plastometer in the 200 to 250 range and be a full 2 in. thick in order to still provide adequate cushioning after the cover has been ground several times. The roll should be mounted on arms with provision for a sufficient range of adjustments. The arms and holders should be lined up with the cylinder mold so that adjustments can readily be made without skewing the couch roll with the mold.

Attention should also be given to the arm mechanism supporting the couch roll since improper pivot location can cause operating difficulties. If, because of framing considerations, the pivot for the couch arm must be located on the out-going side of the couch nip, it must be kept low to prevent formation of a knuckle which can cause excessive loading and damage to the cylinder mold.

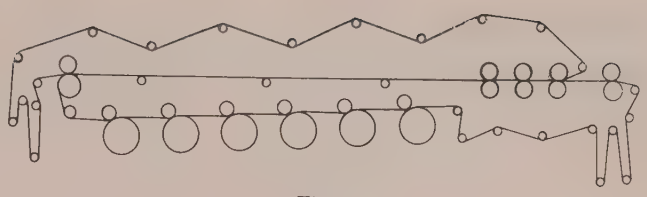

Fig. 4

Assuming that the wet end under consideration is a conventional one (Fig. 4) with a suction return drum press and the bottom felt returning over the vats to the first press, there are several points which must be considered. The modern drum press consists of a large diameter suction roll, usually 30 in. diam., which is equipped with a wide box covering better than $^1/_4$ of the circumference of the roll. The majority of the distance traveled over this box is covered by the bottom felt and the sheet alone, with the top felt being brought into contact at the top of the drum, at an angle of approximately 5° below the horizontal. The purpose of this arrangement is to condition the sheet over the majority of the box area in order to avoid picking problems with the top felt in the liner. A suction slice is usually recommended for the top felt ahead of the drum press nip to help avoid crushing problems. Also, in order to derive full benefit from the drum press installation, the plastometer of the top roll and adjustment for the top roll setting should be picked for minimum crushing.

The final element to be considered is the primary press section. Since development of better draining felts and such modern equipment as suction return drum presses, the pipe roll, baby or squeeze presses of older machines have been outmoded. Today's primary press section consists of heavy cast cores, mounted in antifriction bearings. Incidentally, the present trend is generally away from the overhead primaries above the vats in favor of the more accessible in-line position.

It is believed by some that the felt run through the primary press section must be kept to as near a straight line as possible to avoid checking difficulties, although there are instances in actual practice which tend to contradict this. In any event, couching adjustment of the top roll should be provided so that the nip load can be applied perpendicular to the felt, as well as backward couching for freer draining stock.

The proper number of primary press units should be included to provide graduated weighting of the presses and gradual conditioning of the sheet as required by the grades to be run. Loading of both the suction return drum top roll and the primary units is most likely to be specified pneumatic on a modern machine because of the ease of control and the clean design provided.

Crowning of the primary rolls, as in other press rolls throughout the machine, should be carefully specified to give the best possible compromise for the various nip loading conditions encountered on the several grades run. In this connection, it might also be mentioned that crownless rolls have been applied successfully to primary press sections and that they give the flexibility of operation which would be expected.

In conclusion, we have attempted to present a review of various elements of machine design which affect the operation of a cylinder board machine and its ability to produce the desired sheet of board. These design concepts have been proven in actual installations throughout the country on a wide range of board grades.

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Deionized Water "Salvages" Paper Machine Condensate as Feedwater for High-Pressure Boiler

R. J. BURROCK and J. T. YODER

Deionized water solved the problem of making paper machine condensate suitable for use in a new high-pressure steam boiler at Minnesota and Ontario Paper Co. plant at International Falls, Minn. This boiler, rated at 240,000 lb. per hr., 1290 p.s.i.g., 800°F. superheat, was installed to furnish steam for a 6250-kw. topping turbine and to provide reserve capacity for anticipated future requirements.

The problem arose because, although there was enough condensate from the existing 450 p.s.i.g. steam system to supply the new boiler, the quality of the condensate would be inadequate for use in a high-pressure system. The quality was lowered because paper machine condensate was being added to the boiler condensate. The paper machine condensate, which represented about one-fourth of the total condensate, was produced by quenching the steam at the machines with softened water containing 40 p.p.m. dissolved solids and 2 p.p.m. silica. Due to the solids added by the soft water, the paper machine condensate contained about 25 p.p.m. solids and 1.0 to 1.5 p.p.m. silica. When this was mixed with the boiler condensate, the resulting blend contained about 9 p.p.m. solids and 0.3 to 0.5 p.p.m. silica—well below minimum requirements for a high pressure system.

It would have been impractical to approach the problem by replacing the quenching process with surface condensers or high-temperature condensate pumps because the operation of the paper machines is dependent on the quench water. Furthermore, it was necessary to have a supply of high quality water on tap for boiler feed during plant start-up when condensate is not available or if the condensate should become contaminated. Make-up requirements range between 100 and 300 g.p.m. depending on paper machine consumption and available condensate. The only practical solution to both requirements was to continue to quench, but to use water whose quality would be at least as good—and preferably better—than that of the condensate. A system was installed to provide a continuous supply of high-purity deionized water.

The deionizing system consists of two cation exchanger columns each containing 69 cu. ft. of Amberlite IR-120 cation strongly acidic exchange resin, and two anion exchanger columns each containing 83 cu. ft. of Amberlite IRA-400 strongly basic anion exchange resin manufactured by Rohm & Haas Co., Philadelphia, Pa. A 300-g.p.m. vacuum deaerator is installed between the cation and anion exchange units and the deionized water is stored in a 50,000 gal. tank to be consumed as needed. The present 300 g.p.m. capacity of the water conditioning system can, of course, be increased by installing additional ion exchange units and deaeration facilities. Figure 1 is a flow sheet of the feedwater treatment circulating and condensate systems.

A multiple bed system was selected in preference to a mixed bed unit for this plant to provide better accessibility to the anion resin since the rather high level of organic matter in the filter plant effluent was expected to cause fouling. Other considerations involved lower capital cost per unit of exchange capacity and higher regeneration efficiency for a multiple bed system.

The water supply for the ion exchange units is pretreated in an existing filter plant which reduces turbidity, color and dissolved and colloidal organic matter. See Table I for a typical analysis of the filter plant effluent. The raw water, obtained from the Rainy River is screened, chlorinated and coagulated with alum and clay (plus activated silica in winter) in rectangular precipitators and filtered through sand filters at the rate of 2.9 g.p.m. per sq. ft. This treatment is designed to provide low-color water suitable for pulp bleaching and removes about 60% of the organic matter. The water has a chlorine residual of 0.8 p.p.m. which is removed with sodium sulfite ahead of the ion exchange units.

R. J. Burrock, Chief Chemist, and J. T. Yoder, Analytical Chemist Minnesota and Ontario Paper Co., International Falls, Minn.

Table I. Typical Analysis of Filter Plant Effluent

Bicarbonate (HCO_3)	4 p.p.m.[a]
Chloride (Cl)	4
Sulphate (SO_4)	22
Soluble silica (SiO_2)	2
Free carbon dioxide (CO_2)	7
Calcium (Ca)	18
Magnesium (Mg)	6
Sodium (Na)	6
Iron, as Fe	0.1
Alumina, as Al_2O_3	0.5
Organic matter, $KMnO_4$ consumed, as O_2	4
Turbidity, as SiO_2	0.5
Phenolphthalein alkalinity	0
M. O. alkalinity	4
Hardness	24
pH	6.0
Free residual chlorine	0.8
Color (Standard Pt-cobalt units)	2

[a] As $CaCO_3$, except as indicated.

The deionization system provides water of excellent quality for quench and make-up having a specific conductivity of 1.5 to 3 micromhos and a silica content of 0.005 to 0.015 p.p.m. This quality is obtained with a regeneration level of 7.2 lb. H_2SO_4 and 4.3 lb. NaOH per cu. ft. of resin.

PILOT PLANT

Before the ion exchange system was installed, a pilot plant was set up to obtain data pertinent to the design and operation of a fullscale system. Of particular interest was determining the effect of organic matter, color and turbidity on the performance of the anion resin, which is a common problem with many surface water supplies. The pilot plant unit consisted of two 24-in. ion exchange columns containing 0.075 cu. ft. of Amberlite IR-120 and IRA-400 resin, respectively. The columns had an inside diameter of 3.6 in., a cross section area of 10.2 sq in., and had sufficient height to provide 100% freeboard over the resin bed to handle a 75% bed expansion during backwashing.

A total of 304 water deionizing runs conducted at a flow rate of 2.3 g.p.m. per cu. ft. of resin were made in the pilot plant, representing a cumulative flow of 1,082,000 gal. per cu. ft. of resin. The resins were operated at a regeneration level of 10 lb. of acid (2% solution) and 5 lb. caustic (5% solution) per cu. ft. of resin. After several runs to exhaustion to determine initial capacity of the fresh resins, the columns were subjected to successive sets of 20 runs to 90% of exhaustion. Between each set of 20 runs, several runs were made to the full exhaustion point to obtain data on capacity, specific conductivity, pH, rinse requirements and other op-

Table II. Operating Conditions for Demineralizer Plant

	Cation	*Anion*
Resin (Amberlite)	Amberlite IR-120	Amberlite IRA-400
Resin, quantity, cu. ft.	69	83
Bed depth, in.	30	36
Bed cross section area, sq. ft.	27.6	27.6
Service flow rate, g.p.m.	150	150
g.p.m./cu. ft.	2.2	1.8
Backwash flow rate, g.p.m./sq. ft.	5.1	1.0
Regenerant	66 Bé H_2SO_4	50% NaOH
Regenerant level, lb./cu. ft.	7.2	4.3
Regenerant concentration, %	2,4,6 stepwise	5
Regenerant flow rate, g.p.m./cu. ft.	1.2	0.14
Rinse rate, slow, g.p.m./cu. ft.	1.0 (10 min.)	0.14 (40 min.)
Rinse rate, fast, g.p.m./cu. ft.	2.2 (30 min.)	1.8 (70 min.)

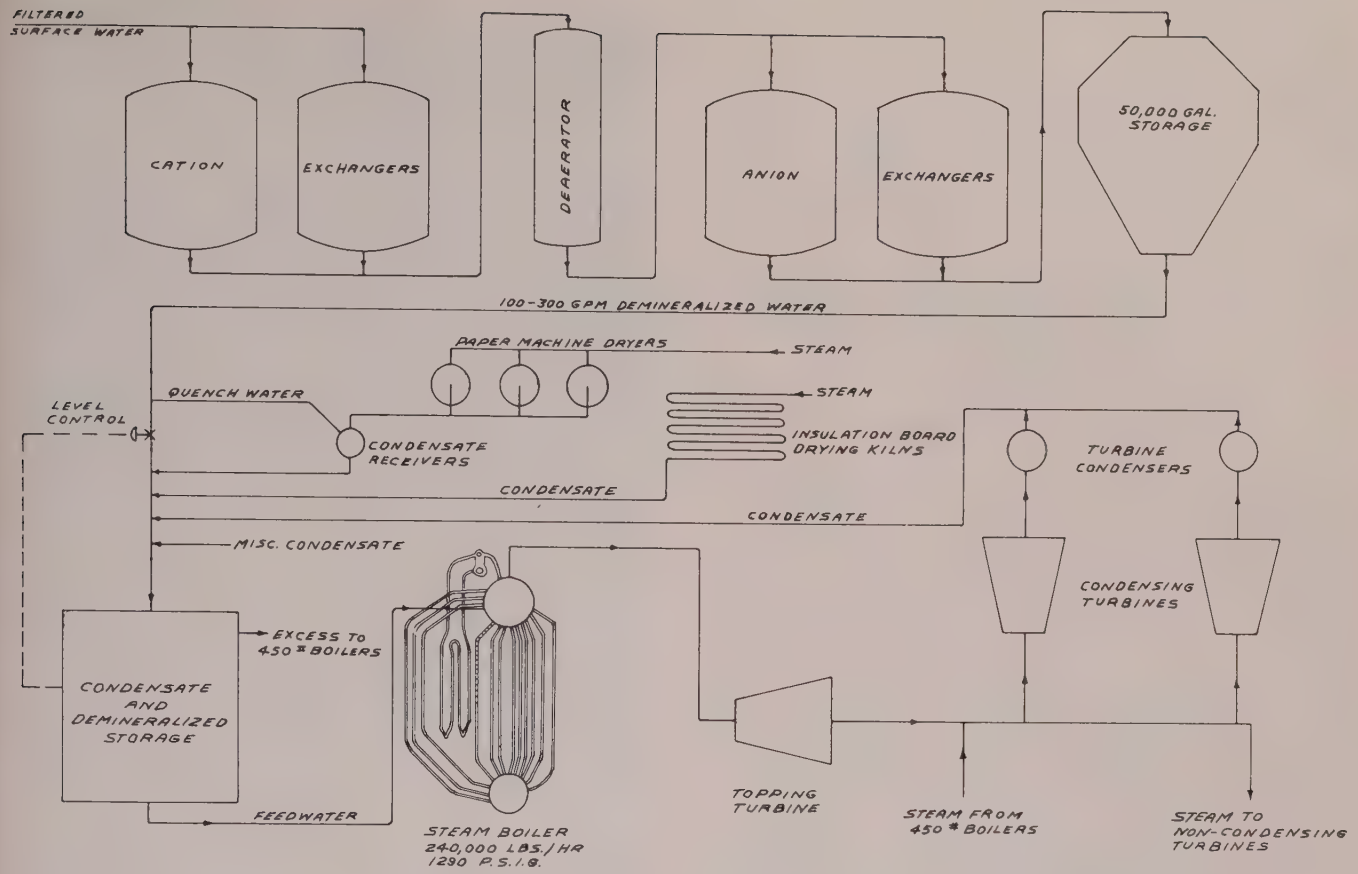


Fig. 1. M & O Paper Co. demineralizer, high pressure boiler feedwater and condensate return systems

erating information. The exhaustion end points were 0.15 p.p.m. silica and 0.08 p.p.m. sodium for the anion and cation resins, respectively.

Procedure for Alleviating Organic Fouling

After 104 cycles, corresponding to a cumulative throughput of 375,000 gal. per cu. ft. of resin, the capacity of the anion exchange column dropped to 73.5% of the initial capacity, presumably because of fouling by organic matter not removed by the pretreatment plant. Breakthrough came after 3300 gal. per cu. ft., compared with 4487 gal. per cu. ft. when the resin was new. In collaboration with Rohm & Haas Co. and Cyrus Wm. Rice & Co., the following procedure was worked out to restore the capacity of the anion resin. First, 10 lb. Nacl (10% solution) per cu. ft. of resin was fed to the resin at the usual regenerant flow rate of 0.5 g.p.m. per cu. ft. This was followed by introducing a solution of 0.5% NaOCl and 1% NaOH which was left in contact with the resin overnight. The resin was then rinsed and regenerated in the normal manner. In this instance the oxidizing treatment restored the exchange capacity to 90% of its original value.

With additional cycles the capacity of the anion resin again dropped, and the oxidizing procedure was employed after every 50 cycles for the remainder of the pilot plant operation, that is, after cycles nos. 155, 205, 254 and 301. After the first treatment, the salt-hypochlorite procedure was modified by adding a third stage of treatment, application of 10% salt solution following the hypochlorite. In all cases the capacity returned to 85 to 90% of the original value, except in the last instance when it returned only to 70%. The amount of capacity loss from fouling and the amount of re-

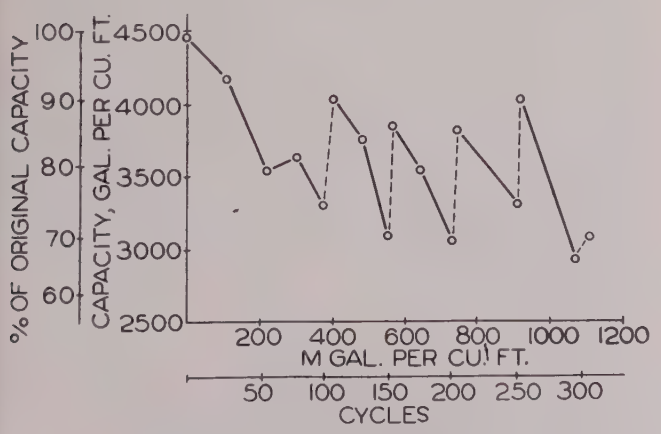


Fig. 2. Anion capacity loss

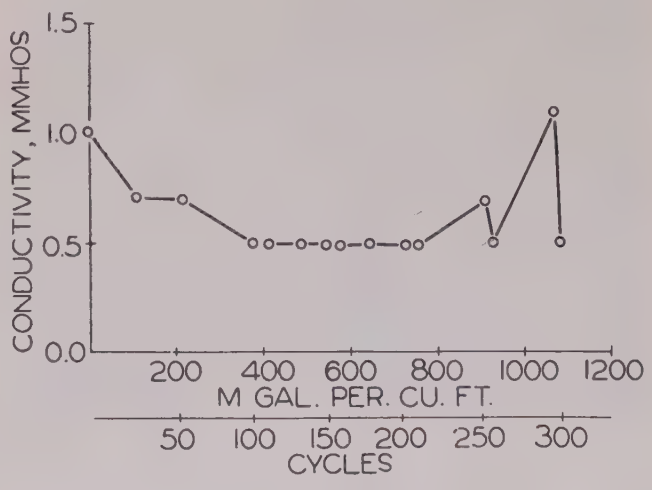


Fig. 3. Conductivity of anion effluent

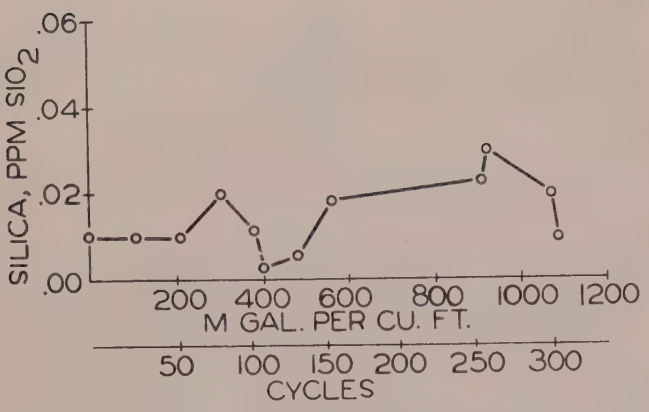


Fig. 4. Silica in anion effluent

covery after each treatment during the 304 operating cycles are shown in Fig. 2.

The results indicate that the oxidizing treatment was quite effective in restoring a high percentage of anion exchange capacity lost due to fouling. This treatment should be applicable to any strong base resin where similar problems are encountered with organic matter in the water supply.

It is interesting to note that despite the decreasing capacity, effluent quality from the anion bed remained consistently high. Specific conductivity averaged less than 1 microhm (Fig. 3), and the soluble silica generally averaged less than 0.02 p.p.m. (Fig. 4), with a large portion of many runs below the instrument sensitivity of 0.001 p.p.m. It was found also that as the number of operating cycles increased, the spread between initial and final pH of the anion resin effluent decreased. During the early runs, the starting pH ranged between 8 and 9, and pH at exhaustion was between 6 and 7. However, after about 200 cycles, this spread reduced to less than 0.5 unit, and the average pH during the runs gradually dropped from an alkaline value and stabilized at about 6.5.

With regard to the cation exchange resin, the pilot plant experience showed that the capacity dropped from an initial value of about 17 kilograins per cu. ft. to about 14 kilograins after during the first 45 operating cycles and then leveled off at that value for the remainder of the 153 cycles during which data was collected. During the last 20 runs the capacity dropped below 13 kilograins, accompanied by a bleaching of the resin. This is believed due to the 0.8 p.p.m. chlorine residual in the filter plant effluent, which is removed with sodium

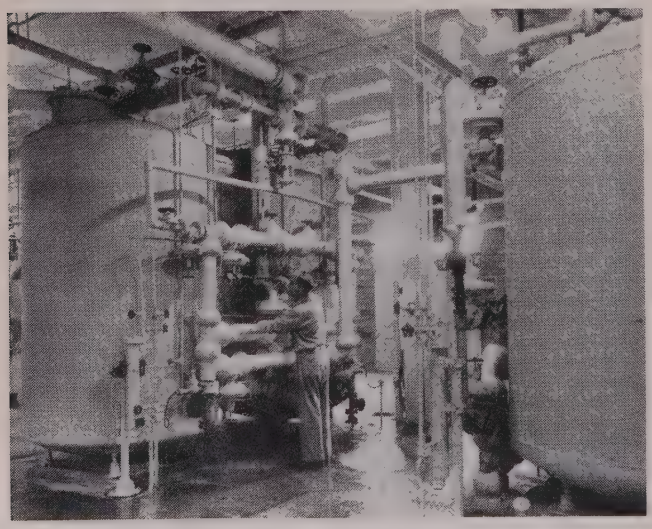


Fig. 5. View of the water deionization equipment. Duplex anion exchanger units are on the left of operating aisle; partial view of duplex cation exchanger units is shown on right

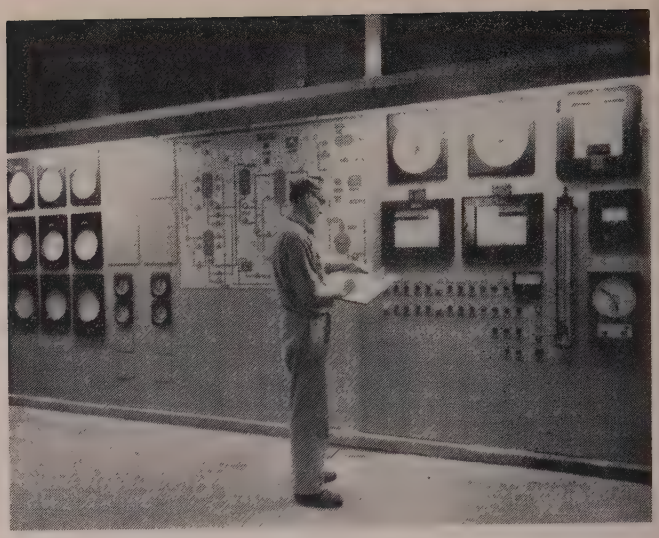


Fig. 6. Instrument and control panel for the deionization equipment

sulphite before the water reaches the full-scale ion exchange system.

Operating Data

By the data collected from the pilot plant operation Minnesota and Ontario Paper Co. was able to determine the exchange capacities of the two resins under their specific operating conditions. For the Amberlite IR-120 cation exchange resin, the capacity was established at about 8000 gal. (equivalent to about 14 kilograins of exchangeable ions as $CaCO_3$) per cu. ft. of resin. For the Amberlite IRA-400 anion exchange resin, the capacity was established at 3000 to 4000 gal. (representing 7 to 9 kilograins) per cu. ft. of resin, depending on the degree of fouling.

In addition considerable experience was gained with techniques of combatting organic fouling, which were extremely helpful in developing the design and operating procedures for the present full-scale deionization system. Furthermore the pilot plant data provided a realistic basis for estimating resin life and operating costs and, by demonstrating that water of consistently high quality could be obtained despite organic fouling, permitted the company to specify minimum quality limits for specific conductivity and soluble silica for the deionized water.

PLANT OPERATING EXPERIENCE

Operating conditions for the full-scale plant are shown in the attached Table II and operating data in Table III.

Regeneration levels for the full-scale plant have been lower than for the pilot plant, 7.2 lb. acid per cubic foot versus 10.0 lb., and 4.3 lb. caustic soda per cubic foot versus 5.0 lb. This change was made to obtain greater regenerant economy. Acid regeneration for the full-scale plant is stepwise at 2, 4,

Table III. Operating Data

	New	*After 2 years service*
Cycles		168
Average capacity, gal./cu. ft.	4760	4220
kgr./cu. ft.	9.52	8.44
% of original	100	89
Average anion effluent conductivity, mmhos.	1.5–3.0	1.5–3.0
Average anion effluent silica, p.p.m.	0.005–0.015	0.005–0.015
Average anion effluent pH	9.8–9.3	8.4–4.7
Average anion rinse requirements, gal./cu. ft.	110	100
Total water treated, gal./cu. ft.		760,000

Note: Three salt treatments have been applied up to 1/27/59, no. 1 after cycle 91, no. 2 after cycle 122, and no. 3 after cycle 146.

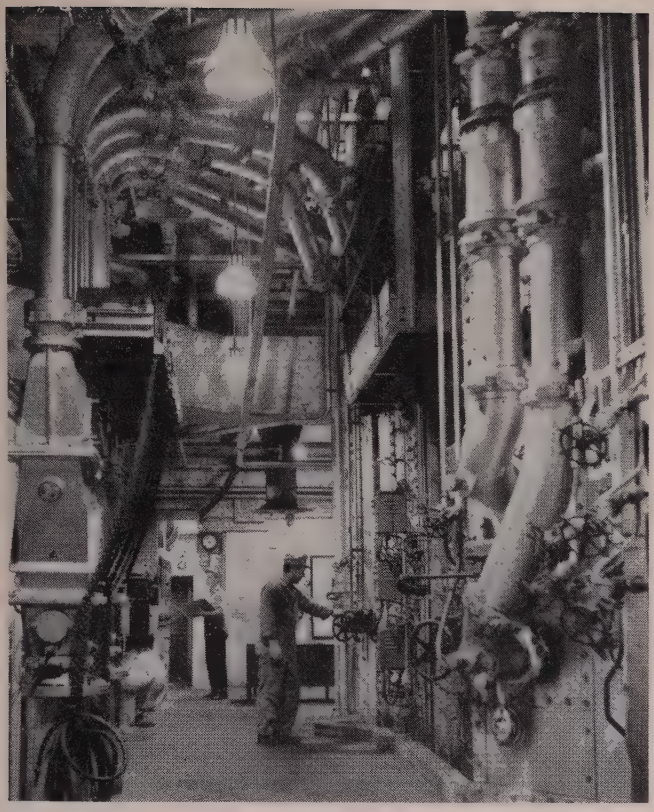


Fig. 7. High pressure steam generating unit arranged for pulverized coal firing—continuous capacity of 240,000 lb./hr. operating at 1290 p.s.i.g. 800 F.T.T. All available condensate returned from plant process is utilized primarily as feedwater for the high pressure boiler, with deionized water makeup added as required

and 6% concentration for greater efficiency. In the pilot plant the acid regenerant concentration was maintained at 2% for reasons of practicability.

The salt treatment has thus far consisted of 10% brine at 120°F. passed through the unit during an 80-min. period, then left in contact with the resin overnight. Treatment level is now established at 20 lb. per cu. ft. of resin. Salt applications are planned for intervals of 20 to 25 cycles. Future treatments are planned with 10% brine-1% caustic soda solutions. The hypochlorite treatment has not been used in the plant units.

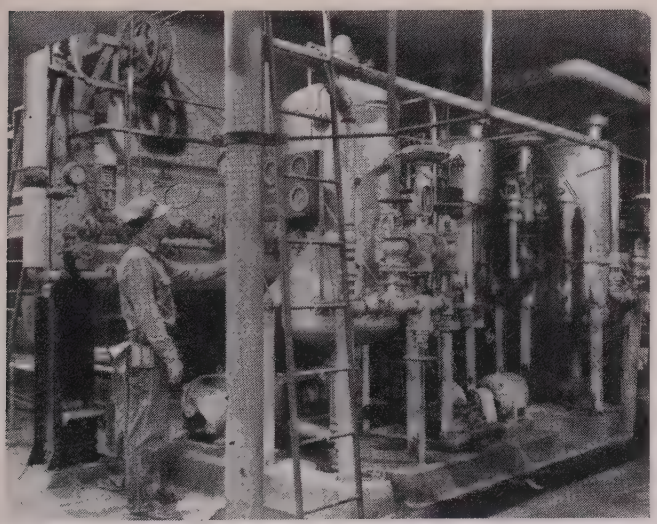


Fig. 8. Condensate drainage system for dryers on no. 3 paper machine. Deionized water is utilized to quench flash steam and reduce temperature of paper machine condensate. View at left shows deionized water supply line with temperature control valve leading to last stage receiver which is equipped with direct spray type flash steam condenser

The three salt treatments applied thus far have restored capacity from about 90% of original to 100% of original. This is better than pilot plant results, and may be due to the practice of treating the resin at 20–25-cycle intervals instead of allowing capacity to fall to 70% before treatment.

Figures 5 to 8 show various sections of the deionization system and the manufacturing plant.

A Study of the Affinity of Direct Dyes for Cellulose

RICHARD ALAN BOSS

In the study of dyeing cellulose, there are many different chemical and physical phenomena which must be considered. The evaluation of the contribution of each is extraordinarily difficult. To add to the difficulty, the dyestuffs used in most cases are not "pure," and they present complex problems of purification. It is not surprising, therefore, that dye chemistry is still to a large degree an empirical art, nor is it surprising that competent investigators disagree as to the basic causes of observed phenomena.

The excellent and comprehensive monograph of Venkataraman (*1*) critically analyzes the work of many investigators who have attempted to explain the dyeing process. It also explains the reasons for apparent inconsistencies among them.

The actual process of dyeing with direct dyes is probably a combination of adsorption and precipitation (salting out). Precipitation in itself will not guarantee that the dyestuff will remain on the cellulose once it is removed from the dyebath, unless there exists affinity between dye and the adsorption medium.

In the case of direct dyes, there seems little doubt that the attraction of the azo (—N=N—) groups of these dyes for the hydroxyl (—OH) groups on the cellulose is the main affinity factor (*2*). From the investigations of Schirm (*3*), there likewise can be little doubt that in order for these attractive forces to have their effect, the repulsive effects of the ionized sulphonate ($—SO_3—$) groups must be overcome.

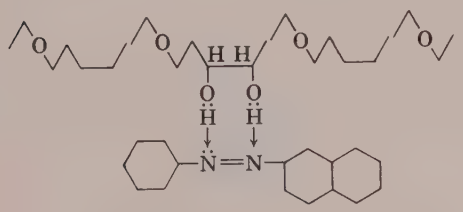
Internal hydrogen-bonding tendencies may be the cause of differing affinities of various direct dyestuffs. Wulf, Hendricks, Hilbert and Liddel, as cited by Pauling (*4*), have reported on infrared absorption determinations on such substances as phenylazo-1-naphthol-2 and *p*-hydroxyazobenzene. From the absence of any significant absorption in 7000 $cm.^{-1}$ region (wavelength units) a strong intramolecular hydrogen bond was postulated for the former substance. The latter substance did not show the same effect, so that any hydrogen bonding present might be expected to be very weak.

Intramolecular hydrogen bonding may interfere with the affinity of a dye molecule of cellulose. If the structure of the molecule is similar to phenylazo-1-naphthol-2, one may expect the intra-molecular effect to predominate because a chelate O—H···N bond may form between the hydroxyl group and the azo group satisfying the requirements of stereochemistry (Fig. 1a). Such a bond is certainly strong enough to diminish the tendency for the azo group to form any other bond with the hydroxyl groups present on the cellulose molecule.

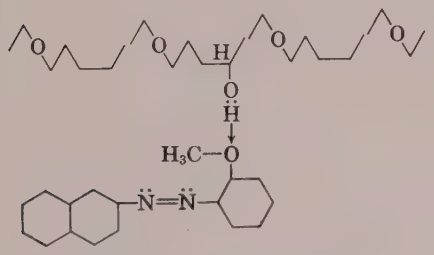
Other types of hydrogen bonding, both intramolecular and intermolecular, which may exist should be mentioned.

Richard Alan Boss, Graduate Student, University of Maine, Orono, Me. (Present Address: Marinette Paper Co., Subsidiary of Scott Paper Company, Fort Edward, N. Y.). Work performed in Department of Chemical Engineering, Univ. of Maine.

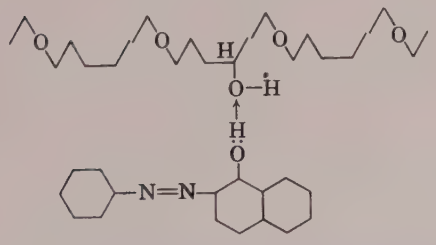
(a) Intramolecular hydrogen bonding (chelation)



(b) Intermolecular hydrogen bonding



(c) Intermolecular hydrogen bonding



(d) Intermolecular hydrogen bonding

Fig. 1. Intermolecular and intramolecular hydrogen bonding

Hydrogen bonding may occur between cellulose and azo groups present on dye molecules (Fig. 1b). Hydrogen bonding may occur between cellulose and ethereal oxygen of a methoxy group on a dye molecule (Fig. 1c). Hydrogen bonding may occur between a hydroxyl group on a dye molecule and a hydroxyl group on cellulose (Fig. 1d). Hydrogen bonding may occur between cellulose and amine groups on the dye molecule (Fig. 2a). Chelate formation may occur between a hydroxyl group in position one and an amine group in position eight in a naphthalene ring (Fig. 2b). The presence of a sulphonate group adjacent to an azo group depending on the pH may cause a zwitterion effect (Fig. 2c).

In the case of a substance such as *p*-hydroxyazobenzene, no intramolecular hydrogen bonding is possible although an association or polymerization between the dye molecules might be possible (Fig. 2d). Such an intermolecular bond if it exists at all would be quite weak.

Many and varied techniques have been outlined for study of the affinity of direct dyes for cellulose. In this investigation chromatography was chosen as the method for research. A vertical chromatographic tube was employed, and pure powdered cellulose was used as the adsorbent medium. The distance traveled down the column by the dye in unit time was considered to be inversely proportional to the affinity of the dye for cellulose.

MATERIALS AND METHOD

A vertical chromatographic tube was chosen to measure the affinity of azo dyes for cellulose (see Fig. 3). The column or tube used in this investigation was 2 ft. in length and 25 mm. in inside diameter.

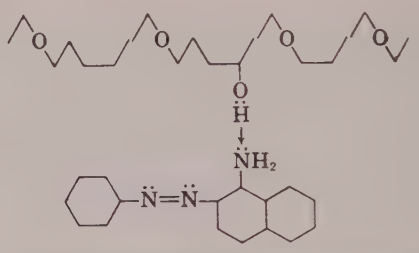
In order to control the drainage rate through the column, a glass stopcock fitted to a cork stopper was inserted into its base. Four small holes were bored into the top of the cork into which were placed 1-in. lengths of 6-mm. tubing. When the cork was inserted into the tube, these pieces of tubing served as a support for a porous procelain disk. The purpose of this disk was to restrict the drainage through the tower to liquid wash water.

Another porcelain disk, suspended by copper wires, was placed on top of the powdered cellulose after the tube had been packed. The porcelain disk provided a protection against disrupting the formation at the top of the column when liquids were introduced. Uniform density and good formation within the column were very important for a successful chromatogram.

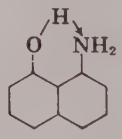
The adsorbent medium used in this experimentation was Solka-Floc, a highly purified powdered wood cellulose. The dyes used were direct azo dyes of varying structures and molecular weights.

The chromatographic column was loaded with 30 g. of powdered cellulose and packed to the desired height. The packing was done carefully so that a uniform density would be obtained thoughout the tower. With the tube packed to a height of 11 in., distilled water was introduced into the top of the tube, and allowed to pass down the tower continuously. This washing was continued for about 2 hr. to insure complete wetting of the cellulose.

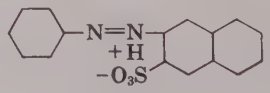
After the allotted time for the wetting had elapsed, 40 ml. of a 2% dye solution containing 5% of 0.025 *N* KCl was



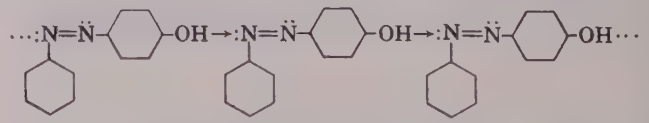
(a) Intermolecular hydrogen bonding



(b) Intramolecular hydrogen bonding (chelation)



(c) Zwitterion effect



(d) Intermolecular hydrogen bonding

Fig. 2. Intermolecular and intramolecular hydrogen bonding

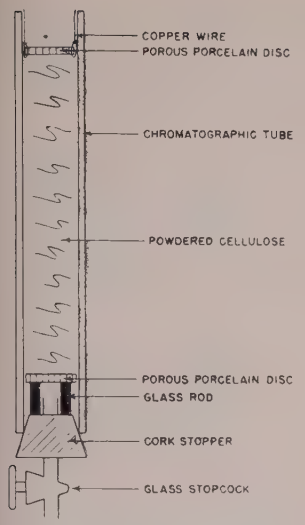


Fig. 3. Chromatographic equipment

introduced into the tower. When all the solution had been adsorbed, distilled water was again continuously introduced into the tube and a 20-min. test period was begun.

During this testing period, measurements were made every 2 min. on the following variables: volume of liquid drained from the column, and the distance which the dye color ring or rings had traveled down the column.

The above technique was used for chromatograms of pure or mixed dyes. Only the amount and type of dye solution placed in the tower were varied from run to run. A 2% solution was always used so that comparable results would be obtained.

In dealing with spirit soluble (water insoluble) azo dyes, the procedure remained the same except for the type of solvent used. The dyes were dissolved in absolute ethyl alcohol, and the wash liquid used to wet the cellulose and to wash the tower after the dye had been adsorbed was a mixture of 50% absolute ethyl ether and 50% absolute ethyl alcohol.

The procedure remained the same in investigating the effect of absence of electrolyte upon adsorption with the exception that potassium chloride was omitted from the dye solution.

The procedure for determining the effect of dye storage concentration on the relative affinity of azo dyes for cellulose varied only in the preparation of the dye solution. The original solution of 2 g.p.l. was diluted 20 to 1 and allowed to stand for 24 hr. before the chromatogram was run.

DISCUSSION OF RESULTS

The results of the chromatographic tests and the structural formulas of the direct azo dyes grouped for comparison in this section may be found by referring to Table I. The dyes which will be compared in what was found to be their decreasing order of affinity are Pontamine Deep Blue, Direct Blue 2B, Brilliant Benzo Blue, Benzoreinblau, and Diphenyl Blue.

Pontamine Deep Blue has a higher affinity for cellulose than Direct Blue 2B. The former traveled 4.90 in. down the chromatographic tower while the latter traveled 5.00 in. Table I shows that the molecular structure of these dyes is very similar, the only difference being the placement of an amino (—NH_2) group and the lack of a sulphonate (—SO_3^{--}) group on the Pontamine Deep Blue molecule.

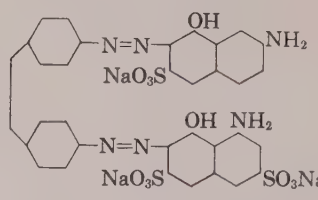
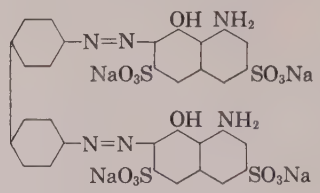
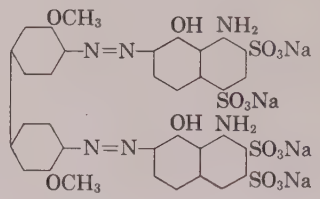
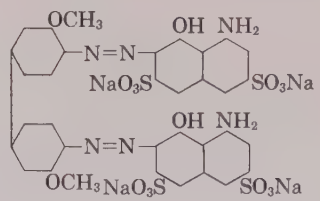
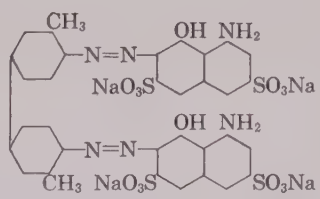
The strong attraction for intramolecular and intermolecular bonding between azo (—N = N—) groups and hydroxyl (—OH) groups was discussed previously in the introduction section of this thesis. In the case of this group of dyes, there seems a definite hindrance for chelate intramolecular bonding to take place between the azo groups present on these molecules and the hydroxyl groups adjacent to them. Such bonding would, of course prevent intermolecular hydrogen bonding between the azo groups and the hydroxyl groups on the cellulose molecule, and thus decrease the affinities of both dyes.

From the chromatographic results of this dye group, it is the opinion of the investigator that the probability of amino and hydroxyl intramolecular bonding is more plausible than hydroxyl and azo bonding. This statement will be explained later in the discussion of the remaining dyes in this group. In a comparison of Pontamine Deep and Direct Blue, chelate bonding between the amino and hydroxyl groups is possible in one position in the former and two positions in the latter. This leaves a hydroxyl group free to bond with an azo group in the Pontamine Deep Blue molecule, thus decreasing the affinity of this dye for cellulose as compared with Direct Blue, by having only one azo group free to bond. However, the tabulated results show that this dye has a higher affinity for cellulose than Direct Blue; this being the case, there must be a stronger effect present causing a decrease in the substantivity of Direct Blue.

Ionized sulphonate groups (—SO_3^{--}) have a strong repulsion effect on cellulose hydrogen bonding. A comparison of Pontamine Deep Blue and Direct Blue dyes offers an excellent example of this effect. Upon examining the molecular structures of these dyes, it is found that Direct Blue has four sulphonate groups while Pontamine Deep Blue has only three. This must account for the main difference in their affinity for cellulose. Direct Blue will presumably have less ability to be adsorbed by cellulose because its sulphonate groups offer a greater repulsion effect for cellulose molecules.

The table of results, Table I, for this group of dyes gives Direct Blue a slightly higher affinity for cellulose than Brilliant Benzo Blue. Direct Blue traveled 5.00 in. down the chromatographic tube, while Brilliant Benzo Blue passed

Table I. Group I Azo Dyes

Name of azo dyes	*Structural formulas*	*Inches traveled in 20-min. period*
PONTAMINE DEEP BLUE		4.90
DIRECT BLUE 2B		5.00
BRILLIANT BENZO BLUE		5.10
BENZO REINBLAU		5.60
DIPHENYL BLUE		5.80

5.10 in. through the tower. Examining their molecular structures, it seems evident that the major differences are the presence of two methoxy groups (—O.CH_3) on the Brilliant Benzo Blue molecule and the arrangement of the ionized sulphonate (—SO_3^{--}) groups.

It is again postulated by the investigator that intramolecular bonding will take place between the amino and hydroxyl groups on both these molecules. In that case, the azo groups are free to bond with cellulose barring restrictions from other sources. In the case of Brilliant Benzo Blue, the methoxy groups present on its molecule ortho to the azo groups will have two effects. First, the methoxy group is an electron donor, and its ethereal oxygen has the ability to form an O—H···O, "hydrogen bridge," with cellulose. Secondly, the presence of this group offers a physical hindrance in the ability of the azo group to form hydrogen bonds with cellulose.

The Direct Blue molecule does not have methoxy groups present in its structure, but the placement of its sulphonate groups, in contrast to Brilliant Benzo Blue, must be considered. The former dye has two sulphonate groups ortho to its azo groups offering some hindrance, by their repulsing effect, for bonding with cellulose molecules.

It seems evident from the results of the chromatograms on these dyes, that the steric hindrance supplied by the presence of the methoxy groups on the Brilliant Blue molecule must offset both its own electron donating ability and the sulphonate hindrance present in the case of Direct Blue.

From Table I, it may be seen that Brilliant Benzo Blue dye has a higher affinity for cellulose than Benzoreinblau. The former traveled 5.10 in. down the tower while the latter traveled 5.60 in. Examining the molecular structure of the two dyes illustrated in Table I, it seems clear that the only difference in their molecules is the placement of the ionized sulphonate groups (—SO_3^{--}).

This comparison is a particularly good example of sulphonate groups ortho to azo groups and their effect on intermolecular hydrogen bonding. The ionized sulphonate group has a repulsing effect on cellulose molecules. Its position adjacent to the azo groups of Benzoreinblau hinders, to some extent, hydrogen bonding between the hydroxyl (—OH) groups on the cellulose molecule and the azo (—N = N—) groups in the structure of this dye. This restriction lowers the affinity of the dye for cellulose thus explaining the reason for the higher substantivity of Brilliant Benzo Blue.

The last dye in this group, Diphenyl Blue, has a lower affinity for cellulose than Benzoreinblau. It may be seen in Table I that Diphenyl Blue traveled 5.80 in. during the chromatographic test while Benzoreinblau traveled only 5.60 in. By referring to Table I, it may be seen clearly that the only difference in the molecular structures of the two dyes is the presence of methyl groups ortho to the azo linkage in the case of Diphenyl Blue while Benzoreinblau has methoxy groups in the same molecular position.

As has been explained earlier, methoxy (—O.CH_3) groups are electron donors. They have the ability to form "hydrogen bridges" with cellulose molecules, and at the same time may offer some steric hindrance to the ability of adjacent azo groups to bond with hydroxyl groups on the cellulose molecule. Methyl (—CH_3) groups, on the other hand, have no ability to bond with cellulose molecules, but their presence ortho to azo linkage still may result in definite physical hindrance to intermolecular bonding.

With these facts in mind, it seems clear that the methyl groups in the Diphenyl Blue structure will lower the affinity of this dye for cellulose to a greater degree than the methoxy groups on the Benzoreinblau molecule.

A very important comparison in the group I dyes may be made among Direct Blue, Benzoreinblau, and Diphenyl Blue dyes. It is an excellent example of the following effects on affinity: first, the absence of any groups on benzene rings ortho to the azo linkage; secondly, the presence of methoxy groups on benzene rings ortho to the azo linkage; and thirdly, the presence of methyl groups on benzene rings ortho to azo linkage.

By referring to Table I of this section, it may be seen that distance down the chromatographic tower increased from 5.00 to 5.60 in. by the presence of methoxy groups, and from 5.00 to 5.80 by the presence of methyl groups. From these results it may be postulated that the steric hindrance effect of the methoxy groups ortho to azo groups has a much greater influence on the affinity of a dye for cellulose than the ability of this group to form O—H···0 intermolecular bond with cellulose molecules.

The postulation presented at the beginning of this discussion section concerning the preferential intramolecular bonding of hydroxyl and amino groups rather than hydroxyl and azo groups was made for several reasons. In the first place, if intramolecular chelate bonding took place between the azo and hydroxyl groups in these various dyes, the relative affinity of each could not be explained to the satisfaction of the investigator. For example, as explained earlier, the only difference in the molecular structures of Brilliant Benzo Blue and Benzoreinblau is the positions of the sulphonate groups on their molecules. The former dye offers no hindrance for intramolecular bonding between the azo groups and the hydroxyl groups in adjacent position. Benzoreinblau has ionized sulphonate groups in a position to oppose such bonding. Attacking the problem this way, Benzoreinblau should have a higher affinity for cellulose than Brilliant Benzo Blue. But the results of the chromatographic tests, tabulated in Table I, show that this is not true.

Secondly, the amino group is more basic than the azo group as may be seen in the action of aniline and azobenzene. It would seem plausible, therefore, that the former would enter into a chelate ring with a hydroxyl group more easily than the latter if equal possibilities for bonding existed for both groups.

The results of the chromatographic tests and the molecular structures of group II dyes may be found by consulting Table II. The disazo dyes which make up this group are Benzopurpurin 10B, Benzopurpurin 4B, and Benzopurpurin B.

The molecular structures of Benzopurpurin 10B and Benzopurpurin 4B are almost identical. The only difference being that the former has methoxy (—0·CH_3) groups on its benzene rings ortho to azo linkage, while the latter has methyl (—CH_3) groups in the same position. Yet, Benzopurpurin 10B traveled 3.20 in. down the chromatographic column while Benzopurpurin 4B traveled 3.70 in.

In the discussion of group I dyes, a comparison between Benzoreinblau and Diphenyl Blue offered an example of the same conditions. As explained previously, the methoxy group adjacent to an azo group has two effects on intermolecular hydrogen bonding. First, the unshared electron pairs of the methoxy group present the opportunity for a weak tendency to form O—H···0 bonds with cellulose molecules. Secondly, this group conceivably could offer a steric hindrance to intermolecular bonding between the azo groups of the dye molecule and hydroxyl groups of the cellulose molecule. The methyl groups on Benzopurpurin 4B and B are not electron donors, but should offer the same physical hindrance to such intermolecular hydrogen bonding.

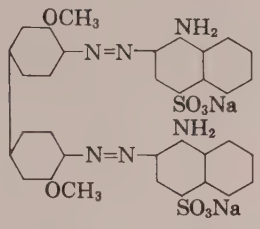
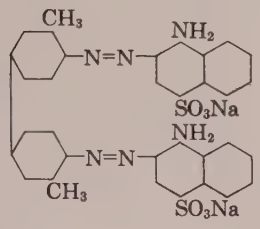
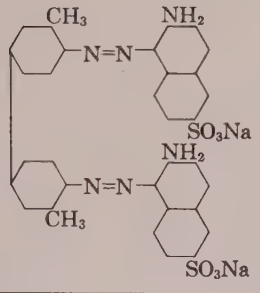
It is to be expected then that Benzopurpurin 10B, because of the effect of the methoxy group and its unshared electron pairs, will have a higher affinity for cellulose than Benzopurpurin 4B. The experimental results show this to be the case.

Benzopurpurin 4B has a higher affinity for cellulose than Benzopurpurin B. It may be seen by referring to Table II that the former traveled 3.70 in. down the packed section of the chromatographic tube while the latter traveled 4.20 in. An examination of the molecular structures of the dyes,

illustrated in Table II, shows that the type and number of groups present are identical, but they differ in their positions in the molecules.

The position of the ionized sulphonate groups present on both molecules and alpha, beta naphthalene attachment seem to be the two variables in question. The effect

Table II. Group II Azo Dyes

Name of azo dyes	*Structural formulas*	*Inches traveled in 20-min. period*
BENZO-PURPURIN 10B		3.30
BENZO-PURPURIN 4B		3.70
BENZO-PURPURIN B		4.20

which each has on the affinity of these dyes for cellulose can only be speculated, and further study is recommended.

Group II azo dyes, generally speaking, have a higher affinity for cellulose than group I dyes. This may be because there are fewer sulphonate groups on these group II dyes. Group I dyes, with the exception of Pontamine Deep Blue, have four ionized sulphonate groups in their molecular structure, while group II dyes have only two. The repelling effect of these groups on cellulose would make intermolecular hydrogen bonding more difficult, and thus account in part for group II dyes having a higher affinity for cellulose.

Group III direct azo dyes contains the following dyestuffs: Congo Red, Congo Corinth, and Azo Blue. Their chromatographic results and structural formulas may be found in Table III.

Congo Red and Congo Corinth have nearly the same structural formula except that, in the former, there are amino and azo groups in each naphthalene nucleus in 1,2 positions. In Congo Corinth, one amino group has been replaced by a hydroxyl group. In the Azo Blue molecule, both naphthalene nuclei have hydroxyl groups in the 1-positions. The results clearly demonstrate that amino groups are very effective in promoting bonding between the dyes and cellulose, and the effect that intramolecular chelate formation has in decreasing affinity.

Congo Red traveled 3.20 in. down the chromatographic column while Congo Corinth traveled 4.60 in. Congo Red has apparently four groups which are free to bond with cellulose. The basic character of the amino groups and their unshared electron pairs afford good opportunities for intermolecular O—H···O hydrogen bonding with cellulose. The unshared electron pairs of the azo groups also offer hydrogen bonding opportunities.

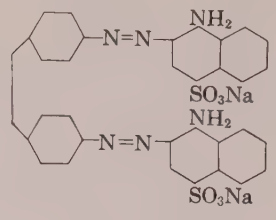
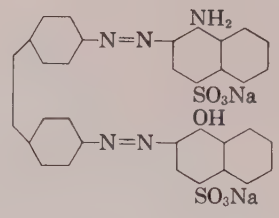
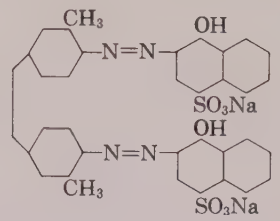
In the Congo Corinth molecule a hydroxyl group has replaced one of the amino groups in position-1 in the naphthalene nucleus. The phenolic hydroxyl group is not only less basic than the amino group of Congo Red, but by virtue of its position it might form a chelate ring with the azo group adjacent to it and, thus, interfere with that group in forming intermolecular hydrogen bonds. This would have the overall effect of decreasing the affinity of Congo Corinth for cellulose as the results indicate.

Congo Corinth has a higher affinity for cellulose than Azo Blue. The former traveled 4.60 in. down the chromatographic column while the latter traveled 5.40 in. In the Azo Blue molecule, both amino groups in position-1 in the naphthalene nuclei have been replaced by hydroxyl groups. It seems clear that the phenolic hydroxyl groups may now be in a position to form intramolecular chelate rings with both azo groups of the Azo Blue molecule, thus noticeably decreasing its affinity for cellulose.

Azo Blue also has two methyl groups ortho to its azo linkage which could offer steric hindrance to intermolecular hydrogen bonding. These groups are not present on the Congo Corinth molecule and may offer another factor in the lower affinity of Azo Blue for cellulose.

Congo Red of this group should also be compared with Benzopurpurin 10B and Benzopurpurin 4B of group II dyes. The molecular structure of the latter dyes may be found in Table II. Congo Red traveled 3.20 in. down the chromatographic column during the 20 min. experimental period while

Table III. Group III Azo Dyes

Name of azo dyes	*Structural formulas*	*Inches traveled in 20-min. period*
CONGO RED		3.20
CONGO CORINTH G		4.60
AZO BLUE		5.40

Benzopurpurin 10B traveled 3.30 in. and Benzopurpurin 4B 3.70 in.

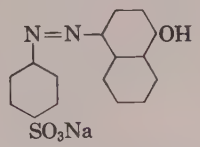
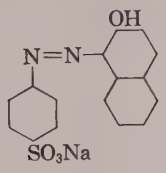
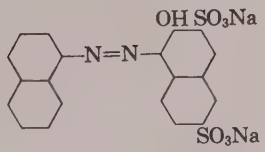
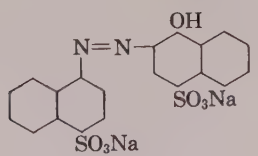
Congo Red and Benzopurpurin 10B have very similar structural formulas. The only difference between the two dyes is that the latter has methoxy (—O·CH_3) groups ortho to its azo linkage. Methoxy groups, as stated previously,

are an electron donating group which has the ability to form O—H···O "hydrogen bridges" with cellulose as well as offering a steric hindrance to intermolecular bonding by adjacent azo groups.

The difference in affinity between these two dyes is very small as can be seen by the results. The conclusion that may be drawn from these results is that the electron donating ability of the methoxy group nearly equalizes the steric hindrance effect upon adjacent azo linkage. In that case, it may be postulated that the methoxy group, in this position on the molecule, will cause only a slight decrease in the affinity of a dye for cellulose. But in the discussion of group I dyes, a similar comparison showed that the steric hindrance had by far the greater effect. This case is not strictly comparable, however, because group I dyes have two sulphonic acid groups on each naphthalene nucleus. These groups, because of their repulsive effects, may emphasize the steric hindrance of the methoxy group while suppressing its weak tendency to accept hydrogen bonds.

Benzopurpurin 4B traveled 3.70 in. down the chromatographic column while Congo Red traveled only 3.20 in. This is another example of the effect of methyl (—CH_3) groups ortho to azo linkage on affinity of dyes for cellulose. These groups present on the Benzopurpurin 4B molecule could offer a steric hindrance for intermolecular hydrogen bonding between the free electron pair in the azo group and

Table IV. Group IV Azo Dyes

Name of azo dyes	*Structural formulas*	*Inches traveled in 20-min. period*
ORANGE I		5.00
ORANGE II		5.30
BORDEAUX RED		5.80
AZO RUBIN		5.80

hydroxyl groups on cellulose molecules. This would account for the lower affinity of the dye for cellulose when compared with Congo Red which does not have groups ortho to its azo linkages to interfere with hydrogen bonding.

Group IV contains four water soluble monoazo dyes studied in this investigation. Orange I, Orange II, Bordeaux Red, and Azo Rubin will be discussed in this section. The tabulated results and structural formulas of this group may be found by referring to Table IV.

Orange I traveled 5.00 in. down the chromatographic column while Orange II traveled 5.30 in. The comparison of these two monoazo dyes offers another excellent example of the effect of intramolecular hydrogen bonding on affinity for cellulose.

Orange I and Orange II have one azo (—N = N—) group, one ionized sulphonate ($—SO_3^{--}$) group, and one hydroxyl (—OH) group. The only difference in structure between the two dyes is the placement of the hydroxyl group. Orange I has its hydroxyl group in position-4 to azo linkage on its naphthalene ring. In this position, there is no chance for intramolecular bonding to take place between this hydroxyl group and the azo linkage. This condition leaves the azo group free to bond with a hydroxyl group present on the cellulose molecule.

The hydroxyl group on the Orange II molecule is in an adjacent position with its azo group, and offers an excellent opportunity for a chelate intramolecular ring to form. This ring will take up the azo group of the molecule and reduce the affinity of this dye for cellulose, because its primary bonding group will not be free to bond with cellulose. As a result of this condition, Orange II will have a lower affinity for cellulose than Orange I. This is substantiated in the tabulated results.

Bordeaux Red traveled 5.80 in. during the chromatographic testing period while Orange II traveled 5.30 in. A comparison of these two dyes is not possible because two variables are present. There are two sulphonate groups on Bordeaux Red and only one on the Orange II molecule. Orange II has a benzene ring in its structure while Bordeaux Red has a comparable naphthalene ring. Any valid comparison of the affinity of the two dyes could not be made.

Chromatographic tests showed that Bordeaux Red and Azo Rubin have the same affinity for cellulose. These dyes each traveled 5.80 in. down the packed section of the column. In both dyes the hydroxyl group is in a favorable position to form an intramolecular chelate ring with adjacent azo linkage. Both dyes have two sulphonate groups in their molecular structure, and it would seem that their different positions is without effect.

SUMMARY OF RESULTS

1. Azo dyes having amino groups show greater affinity for cellulose than dyes of similar structure having phenolic groups in the same positions.
2. Among the dyes studied, those with methoxy groups adjacent to azo groups have less affinity for cellulose than dyes otherwise having the same structure but not containing these groups.
3. Azo dyes having methyl groups adjacent to azo linkage have less affinity for cellulose than those of similar structure having methoxy groups in the same positions.
4. The presence of phenolic groups adjacent to azo groups in azo dyes reduces affinity for cellulose.
5. Azo dyes having sulphonic acid groups adjacent to azo groups show less affinity for cellulose than similar dyes with sulfonic groups in other positions.
6. When comparing dyes of similar molecular structure, the higher the number of sulphonic acid groups present, the lower will be their affinity.
7. Most of the differences in affinity between the azo dyes studied can be explained in terms of intramolecular and intermolecular hydrogen bonding.

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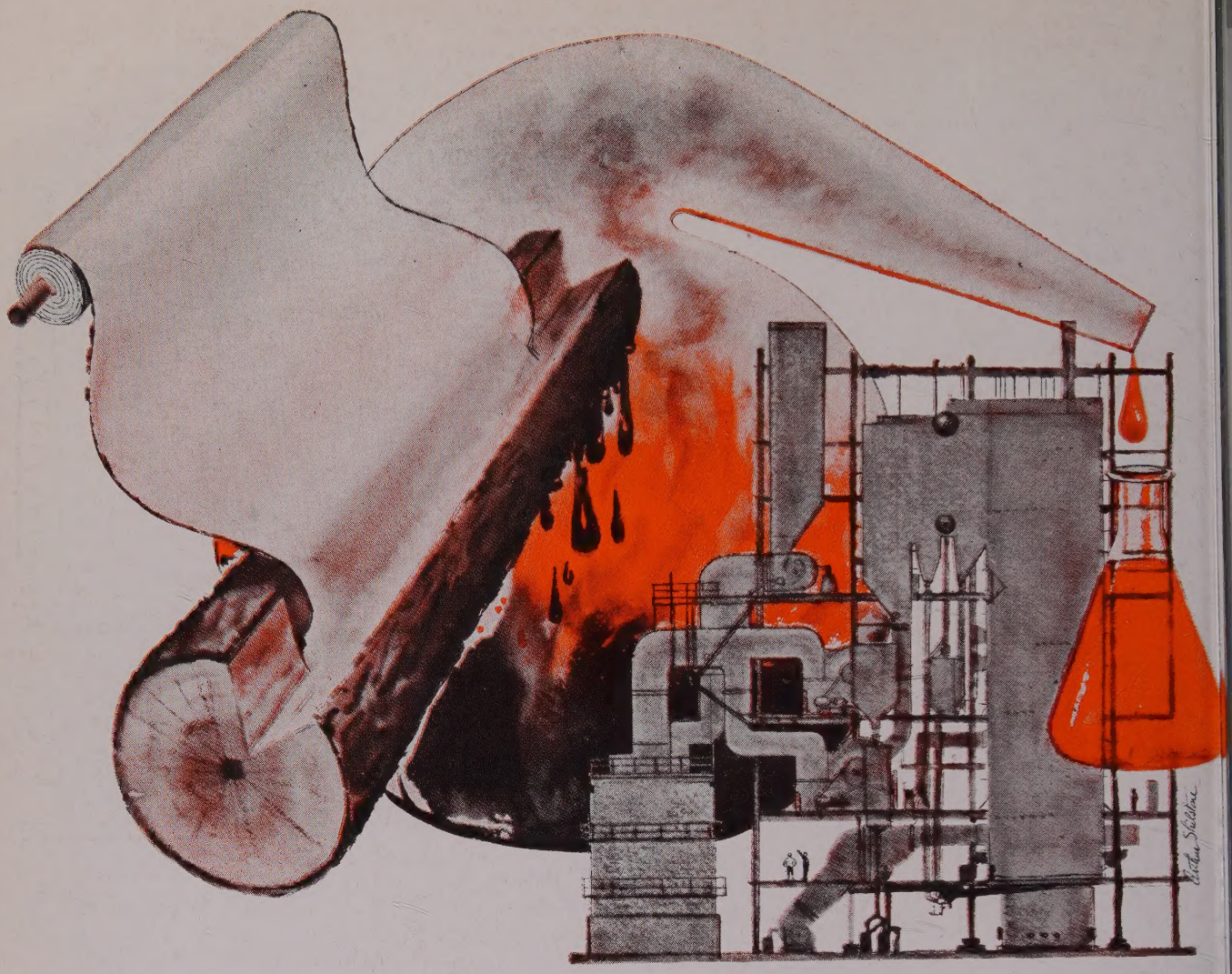

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